

Leverhulme Trust Project.

1: Introduction and background:

The Project is financed by a generous grant from the Leverhulme Trust, and after initial problems in securing staff, is now fully under way. The original aim was to employ a post-doctoral chemist or soil scientist to investigate the chemical nature of the degraded burials at Sutton Hoo, and to understand more about the decay trajectories of the bodies and other buried organic remains. The purpose of this is ultimately to feed the information back into the field, for example to develop chemical indicators to enhance invisible organic residues; and also in a much broader scope, to be able to relate levels of decay to burial environment in a quantitative way, and thus have a predictive index of likely preservation for any terrain encountered in the field.

It was decided after a number of interviews, to put the project under the control of an archaeologist with some scientific knowledge, as the specialist chemist or soil scientist has a training which does not necessarily enable him/her to fully appreciate the archaeological questions being asked. In effect, an archaeologist was appointed to act as coordinator for the scientific analysis of the burials. The project also now includes a research student in the Soil Chemistry Lab. at Birmingham University, who is carrying out some of the analytical work.

The 1986 season was the first full field season at Sutton Hoo in which the Leverhulme Project staff participated, and the main thrust was on recovery of material for post-excavation analysis. The main aim of the sampling programme was to produce a relevant body of material from which comparisons between the natural soil, grave fill, and any organic remains, human or otherwise, could be made, and from which the decay trajectories of the organic remains could be examined. This required a thorough sampling programme throughout the burials, with special care taken to locate the samples accurately.

A beginning was also made to the programme of chemical indicator development, with the first attempts at using a phosphate-presence test for feature location. Subsequent work in the laboratory has involved a trace element analysis of all the samples from one grave, using ICP spectrometry; and amino acid, CHN, polysaccharide and sulphur/chloride analyses on the body samples, to assess the nature of the organic remains. The former is completed, but awaiting final analysis of the results, and the latter is still ongoing.

Other analytical work has been 'farmed out' to other institutions. The main intention with this external work has been to try out various analytical methods, with a view to establishing their usefulness. The results of much of this work are still awaited.

2: Field Work

2.1: Sampling: Introduction.

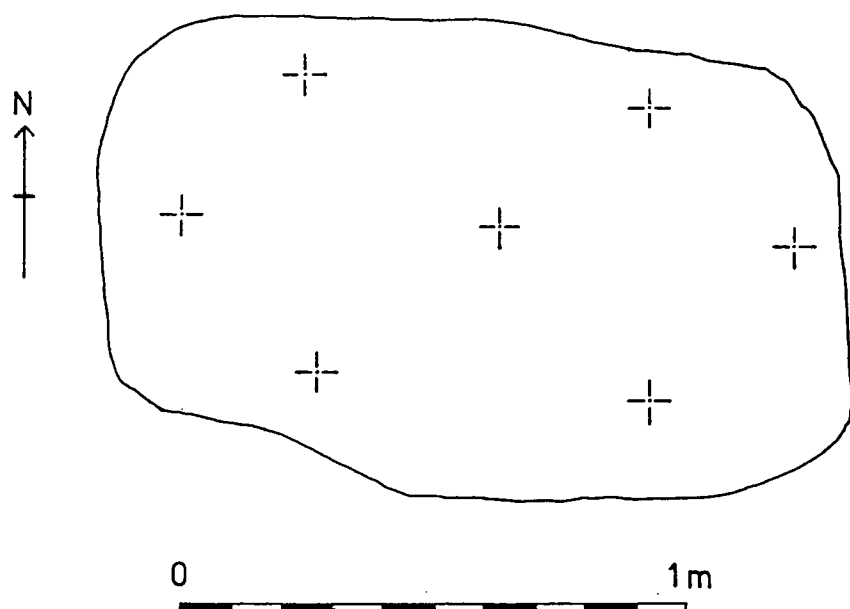
It was apparent that there was an immediate point of conflict between the requirements of the analytical programme and the archaeological record. The normal procedure of archaeological recording requires the maximisation of possible information concerning the position, arrangement and associations of any bodies present. This involves the physical separation of the various components, for example to reveal as much of the body as possible, by removing the fill from around it. In the case of Sutton Hoo, it may well also involve chemical additions to the material, in the form of consolidants. The ideal sampling strategy for chemical analysis would, however, treat the whole grave - fill and body (and any other organic remains, eg. coffin) - as one entity, and seek to create a 3-D chemical picture of the burial, with all the samples equally spaced, and of equal size, and so on. Clearly this would not necessarily provide the archaeological information sought, which still relies primarily on visual assessment of the remains. Only a minute analysis of a grave could approach the level of detail recoverable by standard excavation procedures. It was thus necessary to compromise at the outset, and the sampling procedure was designed to give a fairly even distribution of samples within the grave. In practice, a controlled distribution of samples was possible until an organic object such as coffin or body was encountered. The shape of the object then dictated to some extent the pattern of subsequent sampling. The compromise was such that if the distribution of samples was not completely even, at least the origin of all samples was known (ie. whether they were body, fill, etc.). This was very important for the first set of analyses, in order to characterise the chemical 'signature' of each identifiable type of deposit.

The initial distribution of samples was as shown in the diagram (fig.1a), and the pattern was repeated every 5cm downwards. This would ideally have provided a set of seven columns of samples running from the top through the bottom of the grave. Due to the reasons outlined above, the exposure of the body to reveal the best possible tableau made it very difficult to continue the sample pattern. In fact the body levels show a comparatively larger number of samples, as all the organic decay material was hopefully recovered, but with a distribution that reflects the position of the body, and not the arbitrary positions of the sampling columns. This means that any interpretation of the elemental content of the samples would reflect a more general picture of the elemental distribution than a more strictly controlled sampling programme. The pattern of sampling suggested for future work is shown in fig.1b.

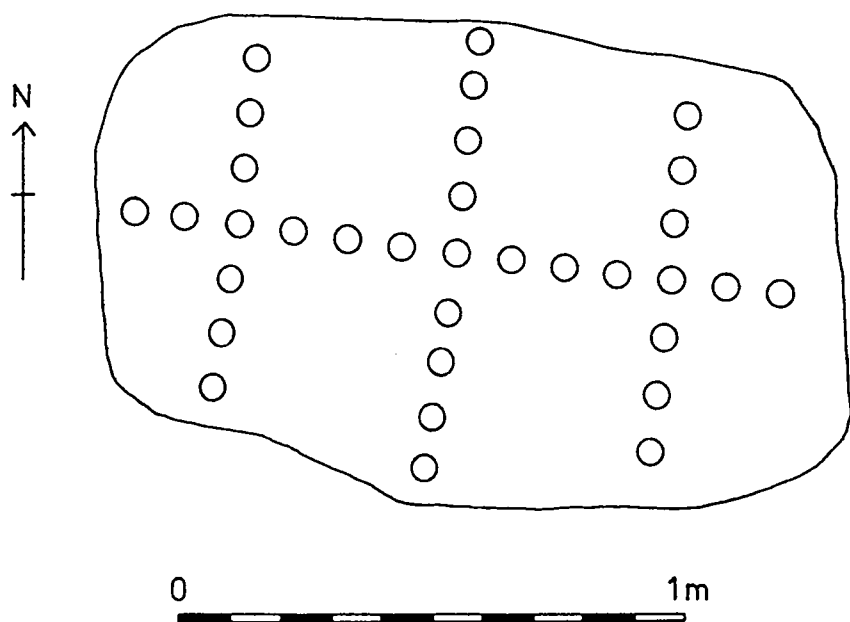
2.2: Sampling: Sutton Hoo.

The samples were taken with a clean stainless steel spatula, and placed in clean Minigrip bags with write-on panels. The sample size varied, because more of the body material was recovered proportionate to the fill. The average size was c. 50g. While taking the column samples,

a) INITIAL SAMPLING PATTERN



b) SUGGESTED SAMPLING PATTERN



care was taken not to dig too deep, so as to merge with the sample below, but rather a larger horizontal area around the point of sampling was removed. This enabled clear vertical differentiation between samples from the same column. Location of the samples was facilitated by use of the site hand-held computers, which were programmed to provide accurate 3-D positional plots from a given theodolite reading, effectively recreating the functions of an EDM. Initial points were carefully fixed, and subsequent points measured from them. The body sample points were generally very accurately fixed, as part of the archaeological recording procedure.

After being taken, the samples went into the finds recording process. It must be said here that they ought to have been air-dried almost immediately, to prevent any post-excavation microbial action taking place, and altering the organic content. However, this proved rather difficult in practice, as there was no room to do this. It remains to be seen whether any samples have been contaminated in this way. They were taken back to Birmingham at the end of the season, and most were immediately deep frozen. There has been a steady programme of drying out and bottling of the samples for long term storage. In future, we would hope to have a drying cabinet on site, and insist that all samples are air dried as soon as possible. Air drying was recommended by Dr. Hayes at Birmingham, as it did not fundamentally alter the conditions under which the samples existed normally, for example waterlogged samples should be kept wet, and so on.

In all, three graves and one suspected grave were sampled, the feature nos. being F226, F227, F231, and F235. Samples were also taken from many of the features in Intervention 39, along with comparative material from the natural around them. It is hoped to be able to analyse these for any differences between the average feature fill and the natural, in order to look into the possibility of chemically mapping the pre-excavation surface. A number of samples from the natural soil around the graves was also taken, and a series of columns from the topsoil through to the natural was recovered (Intervention 43).

2.3: Sampling: Comparative Material.

So far comparative material has been gathered from three other sites in Suffolk, namely Brandon, Hacheston, and Snape.

2.3.1: Hacheston, Suffolk: Suffolk county unit SMR code HCH 013.

The site was visited during the Sutton Hoo season, on 11/08/86. A project representative was conducted to the site by John Newman of the Suffolk Unit, to whom acknowledgement must be given. It is one of a number of sites in the immediate area, around Hacheston and Wickham Market, yielding material from several periods.

HCH 013 was a small area on top of a sandy knoll, which was being removed by sand quarrying. As such it was a rapid rescue excavation, and the detail of recording was correspondingly poorer than one would have liked. The conditions were very sandy, beneath grass cover. I am

not certain whether the area had been under the plough was certainly adjacent to a ploughed field.

The sampling was conducted on a burial identified as pag consisting of a deep grave within a ring ditch (presumably under a small barrow). The grave was approx. 2m N-S x already been partially excavated, the N. end having leave a vertical section (N-facing) across the centre. Samples were taken as a column down the face of this s southern end of the grave was then excavated, and samples coffin stain and horizontally along the bottom of the were no visible signs of an actual inhumation - no remained to show the shape or position of the body. Site spot heights were not recorded, but the sketch plan showed positions of the samples. The site was situated approx 20m AOD contour.

The most noticeable feature of the burial was the evidence of an actual inhumation, although there were v of a coffin, including a large area of the flat bottom traces of grave goods. Chemical examination of the s very interesting, in furnishing proof that this gra inhumation.

2.3.2: Brandon, Suffolk: Suffolk County Unit SMR code

The site, in northern Suffolk, was visited on 3rd Dec project archaeologist. Assistance was greatly appr Suffolk county unit, specifically from Bob Carr and A gave permission for the sampling to be carried out.

Brandon is a large complex site, mainly Middle Saxo settlement, cemetery, riverside and industrial co archaeological record. The conditions are extremely s Hoo, but not apparently as acidic. The site does n been ploughed, so the archaeology, though very shallow undisturbed. The skeletal preservation was much bett Hoo, with the bulk of the skeleton being recognisable visible evidence of soil staining around the body. A stain was visible along the N. edge of the grave exa were, however, rather fragile and difficult to lift. The sampling was done on an inhumation revealed immedi a timber building identified as a church, at the sou current excavation. This in fact lay on the northern already largely excavated.

The burial sampled was that of an adult in extended E-W, lying approximately in the site grid posit 95000/52500. The head lay to the W., and sampling the region of the legs, pelvis, and upper thorax, fro

The burial, and several others being excavated, wer surface of the field, being on average 0.5m below surface. Samples were taken at the E. end of the {

and around the left (?ie. southernmost) tibia; in the pelvic region, and above the upper thorax. Much of the grave fill had been removed so it was difficult to get a continuous column from the top of the grave to the bone. The sample locations are detailed below.

A series of samples was also taken at a point c.10m to the E., from the edge of the excavation, in order to sample the complete profile. 17 samples were taken at 0.05m intervals, from the top of the turf down to the sandy natural.

2.3.3: Snape, Suffolk:

The material from Snape was recovered by the excavators, under the direction of W. Filmer-Sankey, and has not yet been accessioned by the Leverhulme Project. A pagan Anglo-Saxon inhumation with grave goods was sampled.

Sampling procedures carried out on sites other than Sutton Hoo were dictated very much by the prevailing conditions and the time available, and were not as thorough as those carried out at Sutton Hoo.

2.4: Phosphate enhancement spray experiments.

2.4.1: Introduction and Methodology.

The experiment was based on the phosphate spot test described in Eidt, 1973. This involves the use of two reagents added dropwise to a small soil sample on a filter paper, the presence of phosphates being indicated by the formation of a blue phospho-molybdic complex. It was decided to transfer the reaction directly to the ground surface, with greatly increased quantities of reagents, in order to see if any areas of phosphate enhancement would be revealed, ie. phosphate rich features would hopefully stand out as patches of deeper blue coloration against the natural background.

The field procedure was as follows.

Reagent A was prepared on the evening preceding the experiment (6th August), by P. Bethell assisted by C. Williams. The work was carried out in the site office premises, where a small sink and draining board area was available (normally used for photographic work). Reagent A consisted of 290 ml of 10N Hydrochloric acid (HCl), made up to 2.5l with distilled water, and having 95g of ammonium molybdate dissolved in it. The container used was a 2.5l capacity Killaspray (Model no. 4036 Courier 4). Reagent B was mixed on the morning of the experiment, and consisted of 12.5g of ascorbic acid dissolved in 2.5l of distilled water, in an identical container to that used above.

As regards the ease of preparation, the following points were noted. The ammonium molybdate and ascorbic acid were brought to the site in pre-weighed portions, in labelled glass bottles, so their preparation presented no problem on site. The hydrochloric acid was in 2.5l Winchester's, at a concentration of 10N or 36% w.w. In retrospect this

should not have been used. Although safety gloves and goggles were worn, the fumes proved to be extremely irritant, and simple nervousness at handling a potentially dangerous chemical was also a factor. In future, the use of much more dilute acid would be recommended, bearing in mind that this would increase the volume of material to be transported and stored. As a further point, extreme accuracy of measuring the acid was not necessary, as the large volume of reagent used gave some leeway regarding concentration.

In general, the preparation of the reagents was not difficult to carry out, and the limited facilities available were adequate. Assistance was required with the handling of the concentrated acid, but one person could comfortably manage the procedure if dilute acid was used. For safety reasons, however, such work should not be carried out by totally inexperienced people, nor in the absence of a source of running water. Great care should also be taken in the storage and disposal of any chemical reagents. Excess of distilled water should be provided, as it was needed in large quantities for washing the equipment, prior to use. Safety goggles, gloves and lab. coat should be worn.

2.4.2: Field Experiment 1:

7th August 1986

The application of the reagents to the excavation surface was carried out on the morning of 7th August 1986, before the normal working day commenced, in order to minimise any risk of wind-borne contamination to the staff. Present were PHB, MOHC, AC, and NMCB. The area chosen for the experiment was within the stripped and trowelled area of Intervention 32, around F225, F226, F227, and F228. These features were visible after normal trowelling and cleaning, and after spraying with ordinary water, and appeared to have outlines typical of other features which had been found to be inhumations. Thus a phosphate enhancement could be expected if these were inhumations, and thus they ought to show up as blue against the natural sand, when sprayed.

Reagent A was sprayed over the area around these features, as evenly as possible. Reagent B was applied 30 seconds later, again with care to get as even a spread of the reagent as could be achieved. Two people were required for this, both wearing overalls and rubber gloves. A problem was encountered early on, as one of the Killasprays had a faulty seal, and so could not produce as much pressure as the other one. It was attempted to compensate for this during the spraying.

At first, it appeared as if the area had simply been sprayed with ordinary water, the only sign of anything happening was the appearance of a strong blue colouration around the nails fixing the context labels to the ground. Gradually, a blueish/greenish tinge could be discerned, particularly in the feature furthest to the west (F226), which we felt was enhanced by the reaction, and showed the strongest greenish tinge. The others were not so marked, but at the peak of the colouration intensity, c.10 - 15 minutes after the spraying, a clearly green colour was visible over the whole sprayed area. (After c. 10 minutes, the area was sprayed with plain water, which appeared to enhance the colour.

MOHC suggested that future attempts should begin with a thorough wetting of the surface).

Four hours after spraying, the outlines of the graves were still clearly visible, and carried a strong greenish tinge. At 5 p.m., (9.5 hours after application), the stains were still strongly visible, even after rain showers in the afternoon. On gentle trowelling, it was found that the staining reaction affected only the very surface of the soil, ie. <1mm of the feature fill. Because of this, it was very easy to trowel away. The stains remained visible throughout the next day (8th), but were fading. The colouration greatly facilitated the planning of the features.

The experiment was not an unqualified success, but was quite encouraging. A small patch of the anti-glider ditch and adjacent soil in Int. 39 was also sprayed, but with no visible effect.

2.4.3: Field Experiment 2:

9th August 1986.

This experiment was a direct repeat of FXP1, using the same preparation techniques, and spraying over the same area. It was hoped to make this second run a more controlled version of the first, with more careful timing of the colour changes, and so on.

The area of the features already sprayed was fine trowelled to reveal the natural colours once more. This was followed by a fine spray with ordinary water, which was observed to enhance the outline of the features, and also the fine detail of their surface, such as tip lines etc. This began to dry out after only a few minutes, and a second spray with water was carried out before the reagents were applied.

On spraying with the two chemicals, as before with a 30 second gap between the two applications, a greenish-blue tinge appeared almost immediately, but was somewhat patchy over the whole sprayed area, and did not bring out the outline of the features. After two hours, the whole sprayed area was a dark bluish colour, without any distinction between the features and the surrounding soil. The area was sprayed again with water, but this did not aid the differentiation.

This version of the experiment was clearly a failure, the reason apparently being contamination of Reagent A inside the Killaspray. The cause appeared to be a section of metal pipe incorporated in the spraying tube, which was observed to carry blue stains inside it. This indicated that the blue stain was a result of complexing with some constituent of the pipe metal before it even reached the ground. Whether this had affected the results of FXP1, was not certain, but it was felt that on balance the contamination had only become noticeable during the second run.

2.4.4: Field Experiment 3:

12th August 1986.

It was decided to conduct essentially the same experiment over a larger area, to see if any features not visible after trowelling might be

revealed. The Reagent A contamination problem was circumvented by using a different type of Killaspray, the capacity No. 4075 Polyspray 2. This model does not have a pipe attachment of the other, and so the reagent does not come in contact with any metal parts. The area sprayed was Intervention 39, measuring 8m x 4m., before any features were revealed. As before, the reagents were applied at a 30 second interval (water spraying), but it was difficult to get an even coating of reagent on any one spot, because of the difference in pressure between the two Killasprays, and hence differences in pressure, etc.

After 10 - 15 minutes, green-blue streaks began to appear, the effect probably being caused by the uneven spraying - the effect was more pronounced in the darker area. One suspected grave was apparently darker than the others, and BN reported that F24 was clearer than it had been before. At the edge of the lane, an apparently barren patch carried an orange-brown stain, which was felt might indicate a previously undetected feature. After 1 hour, the W. end of the lane was sprayed again with the reagent. A 1 - 2m strip in the centre was resprayed with the reagent. The result in this strip became a much darker blue, and covered most of the area. It did not appear to enhance any features. In general, there was very little clear differentiation over the whole lane.

A series of samples was taken over the whole of 39/Lane 39 grid, with the intention of carrying out a laboratory phosphate analysis over the same area as the spray experiment, for comparison.

2.4.5: Discussion.

It was felt at the time that the phosphate enhancement results were a little disappointing, and that the results did not show anything. However, following the excavation of the features FXP1 and 2, and Lane 39/3D (FXP3), some positive results were revealed. F226 did not contain any visible human remains, but turned out to be a double burial. F228 was not excavated. Int. 39 contained no graves, and in fact most of the features were rather amorphous, and interpreted as natural features. It seemed that the spraying enhanced the anthropogenic features. The negative enhancement shown by Lane 39/3D was in fact a reflection of the nature of the archaeology present. It is in mind that this was by nature a trial experiment, but the results are that it could be very useful in feature detection. F235 shows a phosphate enhancement at the level of the grave fill, but whether backfilled or not is not certain. The phosphate analysis of the samples from Lane 39 has yet been carried out.

What the experiment did show, however, is that it is possible to carry out such spray experiments in a field situation. It can be prepared off-site, or failing that, only limited equipment required for on-site preparation. Providing full safety measures taken, such experiments could be undertaken by anyone with a basic knowledge of handling chemicals. The cost

was not very great either, the most expensive items being the Killasprays.

2.4.6: Conclusion.

The experiment can be regarded as a qualified success, in that the results from two areas of differing feature type and arrangement were different, the area of apparently positive enhancement being found to contain at least one grave, the area showing negative enhancement apparently devoid of anthropogenic features. At the very least, the use of such reagents simply to stain the surface of known features, in order to delimit them in such poor digging conditions as Sutton Hoo, would be useful. The blue stains were observed to last for some considerable time (at least 48 hours), and to only penetrate the upper 1mm of soil. The normal drying rate (under the prevailing conditions) for water-sprayed enhancement is minutes, rather than hours.

In general the experiments were a useful step towards the wider use of chemical enhancement techniques on archaeological sites.

Ref: Eidt, R.C. 1973:

A rapid chemical field test for
archaeological site surveying.
American Antiquity 38, 206-211.

2.4.7: Equipment and reagents used:

1 x 1000ml graduated glass measuring cylinder.
1 x 100ml graduated polypropylene measuring cylinder.
2 x 2500ml polypropylene beakers.
1 x 15cm glass funnel.
1 x glass stirring rod.
2 x 2500ml capacity Killasprays (No. 4306 Courier 4).
Safety gloves (heavy duty rubber).
Safety goggles.

Reagents:

10N Hydrochloric acid (HCl).
AR Ammonium molybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$)
AR Ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$).
Distilled water.

3: Laboratory Analysis: External Analyses.

3.1: Introduction.

Analytical work has been carried out on a number of samples, both 'in-house', and by other institutions. The external work has in general been done on material from earlier interventions, and covers a wide range. The description of that work precedes a report on our own lab. analyses, with the exception of some of the analysis carried out at Queen Mary College, London, which is directly relevant to the major experiment conducted by the project research student.

3.2: Current external analyses.

3.2.1: British Museum.

The British Museum has of course been involved with the site of Sutton Hoo since its original excavation. Although no longer directly responsible for the excavation, the Museum has been assisting the current project with technical and scientific back-up. Their first involvement with material from the recent excavations was an attempt to date a bone sample from one of the burials. The bone was found to contain what seemed at first to be unidentifiable organic matter. After consultation with the Oxford Radiocarbon Accelerator Unit, it was discovered that these were amino acids probably derived from the original bone.

The most recent work undertaken by the Museum for the Sutton Hoo project, is the development of a suitable consolidant for use on the site. This would enable (hopefully) excavation of otherwise unrecoverable fine details of organic artefacts, such as ships, burials, and so on.

This work is currently being done, and no final results are available at the time of writing.

3.2.2: Oxford Radiocarbon Accelerator Unit.

As mentioned above, the Oxford Unit was involved with the original extraction of organic residues from the bone, an important step in its own right, as it shows that organic material does survive under the Sutton Hoo conditions. Whether the case of bone is a special one, in that organic material not part of an integrated organo-mineral complex such as bone does not survive, we have yet to determine fully.

The particular bone sample submitted to the Accelerator Unit yielded 65 mg of amino acids from 200 mg of bone. This was reported to be a "high collagen yield", and "very satisfactory", (Dr. J. Gowlett, pers. comm.). The C-14 date given by the bone sample was AD 750 \pm 70 years, (OxA - 819). This indicated, along with another date from the Harwell lab. of AD 620 \pm 90 (HAR-6800), that the flat-grave burials were roughly contemporary with the ship burial, and could certainly be assigned to

the early medieval period. This was simply confirmation that we should be looking at comparative material from sites of roughly that period, in order to eliminate the time factor in the monitoring of the diagenetic processes affecting the burials.

The Accelerator Unit will become more involved in the work of the Leverhulme project in the near future. It is hoped to send a quantity of extracted humic material from body silhouette samples for analysis. Most usefully, this material will be dated, thereby giving us an indication of whether it is contemporary with the burial (ie. the bones). This will tell us whether the body "stain" is formed of material directly derived from the inhumation, or from subsequent build up of biota decay products over a longer period. This work will be carried out as part of one of the Oxford lab.'s own research programmes, by Dr. Ian Law.

3.2.3: Manchester University.

We have submitted a section of pelvic bone from one of the Intervention XX graves to Neil Garland, a post-graduate researcher in the Department of Rheumatology at Manchester University. His particular field is histology, a biological discipline whose usefulness to archaeology is being widely studied. The techniques largely involve the examination of bone thin sections, with the use of various dyes and light sources to investigate the microscopic variations in the bone tissue. By comparison with known examples, it can be seen how the bone ultrastructure has been affected by the conditions of burial, and it may be possible to deduce such information as relative length of burial, age at death, and sexing within the cemetery. One of the techniques under investigation currently in the archaeo-histological world is the counting of osteons in specific bone locations, as a means of determining age at death.

We are still awaiting results from Mr. Garland, but are looking forward with great interest to viewing his findings.

3.2.4: University College Cardiff.

The most recent involvement with an external body that we have undertaken has been with Dr. Mark Pollard of the Dept. of Chemistry at UCC. Dr. Pollard teaches partly within the Dept. of Archaeology, and is supervising a number of students working on archaeologically-related chemical studies. One such is an experiment to examine the organic residues from an inhumation from a very sandy burial site at Atlantic Trading Estate, Barri, South Glamorgan, of early medieval date. It is hoped to be able to extract cholesterol from "body stain" material recovered from directly beneath the bones, using chromatographic methods. The UCC students have also carried out thermogravimetric analysis on a transect of samples across the body area. The purpose of this is to examine the gross organic content of the soil by monitoring the weight loss during heating to high temperatures. The data should indicate the distance to which the decaying organic "fallout" from the body affects the surrounding soil. We have submitted a number of body

samples from Sutton Hoo, in order to have similar analyses carried out on our own material. The results will provide a useful comparison with material from a similar burial environment, but where skeletal preservation is quite different to that at Sutton Hoo.

We have also been in contact with the Glamorgan-Gwent Archaeological Trust, who are responsible for the excavations at Atlantic Trading Estate, with a view to taking our own samples from one of the inhumations.

3.2.5: Queen Mary College, University of London.

During the course of the project, much help and advice has been received from Dr. Alan Hart of the Dept. of Chemistry, Queen Mary College, London. To assist partly with his own researches, and partly with ours, a number of samples were submitted to QMC for various analyses. These samples had been dried and stored for some time in boxes in BUFAU, and part of the reason for submission was to see if the storage had affected them adversely, eg. by microbial action.

Dr. Hart carried out the following analyses:

'Magic angle' NMR probe of the C-13 spectrum of one of the bone samples.
Conventional solution NMR of the hydrolysed amino acids.
ICP spectroscopy of the samples, and comparison with fresh bone.

These analyses were originally part of the research programme of a post-graduate student under Dr. Hart, who unfortunately has abandoned his studies before completion. Hence the results from these experiments are as yet incomplete, and what results there were have not been followed up. However, some useful and valid conclusions can be drawn from the work done so far.

Analytical results from samples submitted to QMC:

As yet, no results are available for the conventional solution NMR of the hydrolysed amino acids.

The magic angle spin NMR results were not particularly clear, as the spectrum studied, that for C-13, was rather weak. However, the spectrum did seem to show a distinct chemical modification over the spectrum from fresh (bovine) bone. Further work is required to increase the carbon density, by compression of the porous material, in order to get stronger spectra. This is an area of study that would be worth investigating further, in order to understand more about the trajectory of change of specific components of the bone, during the taphonomic processes operating on it.

the results of the ICP analysis are discussed below, in the 'In-house' analysis section, along with our own ICP results.

4: Laboratory Analysis: 'In-house' Work.

4.1: Introduction.

The analytical work carried out by J.Miles, the project's chemical research student, has followed two major directions, that of attempting to extract and characterise humic acids from the body samples, and a trace-element analysis of all the samples from one grave. A series of samples from grave F235, the crouched burial on the north edge of Intervention 32, including wood, body, and grave fill material, has been investigated. It was decided to concentrate on this burial, because it was the only one so far excavated which is known to have no consolidant contamination. It seems that the consolidant spray (PVA emulsion) does not significantly affect the elemental composition of the samples, but it is not yet certain how it alters the results of any analysis of the organic material in the graves. It was felt better to examine a non-consolidated feature in order to compare the results with those from contaminated burials.

4.2: Description of the burial.

Grave F235 appeared on the surface as a sub-rectangle approx. 1.40m x 0.90m. on excavation, it turned out to be c. 0.70m deep, with vertical sides and a flat bottom. It contained the remains of a wooden coffin, F236, and an inhumation, F240. The extant coffin remains were discovered overlying the body.

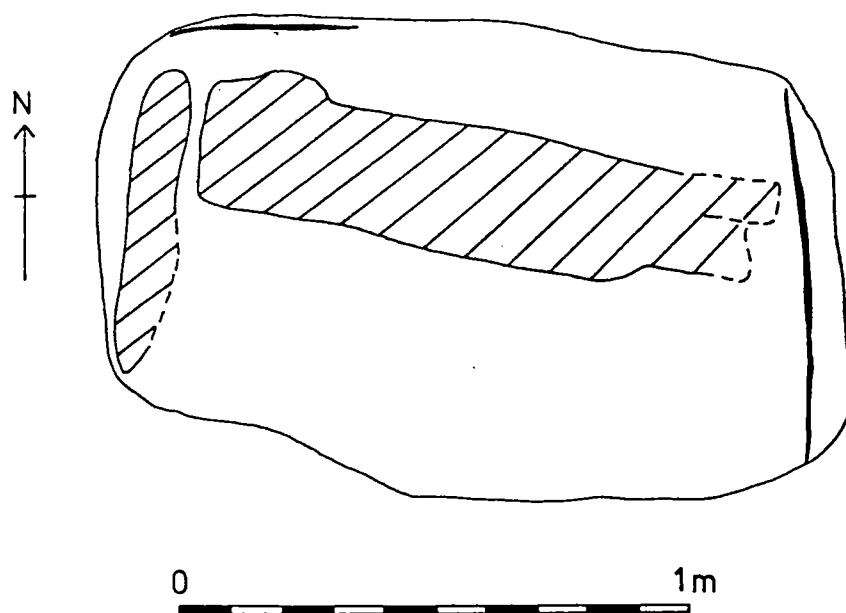
The artefact identified as a coffin was observed as a black staining in the the much lighter sandy soil of the grave fill. It first appeared as mottled grey smudges, becoming darker and more charcoal-like towards what was the centre of the wood. The remains were frequently penetrated by small rootlets, although there was no evidence of larger root systems in the area. The dark stain was excavated to reveal a narrow linear shape, with parallel sides, interpreted as a plank. Traces of other pieces of planking could be seen at the ends of the grave, and the general shape of the wooden remains appeared to be a rectangle slightly smaller than the dimensions of the grave cut.

The body was revealed after removal of the wood traces, and could be clearly seen to be a crouched inhumation, with all limbs traceable. The remains again appeared as staining in the sand, but with a dark brown rather than a black colour, and again with much penetration by rootlets (which in fact helped to preserve the body shape). No bone traces apart from tiny fragments could be discerned. There was also no trace of other organic artefacts such as shroud or clothing, nor any inorganic grave goods.

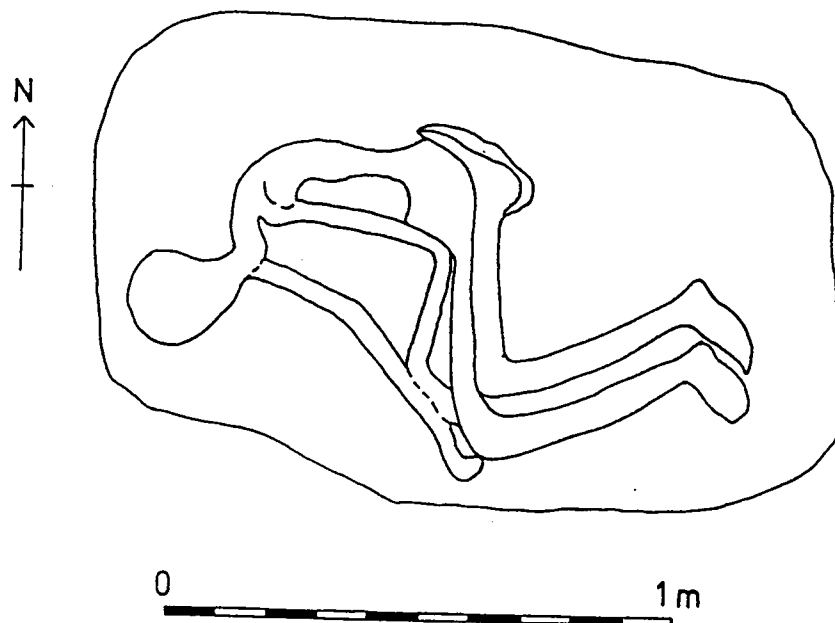
4.3: Humic acid extraction.

The initial experiments were designed to extract humic material from samples recovered during the 1986 field season. The underlying theory is that extraction of the humic material, followed by identification of the various fractions within that material, would give us a clear insight into the origin of the humic substances, ie. whether or not they are

a) LOCATION OF WOOD REMAINS F 236



b) LOCATION OF BODY F 240



directly body-derived. It could also give us some idea of the nature of the bonding mechanism which fixes organic material at the site of burial of various organic artefacts.

The initial extraction has been completed, and a humic acid content of 2.50% has been recorded for the body samples, as against 0.67% for the grave fill material. Work is now under way to extract larger quantities of humic and fulvic acids for further analysis.

Also under way in the Dept. of Chemistry, is an initial organic analysis of the Sutton Hoo material, with amino acid and CHN analyses being currently done.

4.4: Trace element analysis by ICP.

4.4.1: Introduction.

The other major experiment has been an elemental analysis of all the samples from one of the graves, again F235. At this point must also be included an initial ICP analysis carried out for us by Dr. Alan Hart, of Queen Mary College, University of London. This was done as a pilot study to see if the method was useful.

The analysis was carried out on the basic assumption that the elemental composition of a buried artefact is different to that of the surrounding burial environment (ie. soil). On decomposition of the artefact, many of its components are liberated. These liberated elements may be differentially retained in the soil by adsorption onto clay particles, complexation with humic substances, etc.; or they may be leached out of the burial horizon, or used by soil biota in biological processes. The elements which remain in situ may be present in a quite different ratio to that found in the original artefact. Study of the elemental composition of the artefact-affected soil can only fully elucidate these processes in conjunction with other types of analysis, but even so the elemental composition might indicate the nature of the buried artefact, allowing for example the development of element-specific dyes for use in the field.

The elemental composition of the grave fill can act as an important indication of the movement of the soil material occurring since disturbance and burial of the artefact(s). Because the soil system is not uniform the contours of the elemental composition may not be uniform, for example they may follow the contours of different horizons within the soil profile, which need not be horizontal. In order to study the changes in the soil brought about by burial of an artefact, it is essential to identify samples which come from the same 'contour of composition'. For example, where the soil horizons are dipping, a horizontal sampling transect could show up differences which could be wrongly attributed to an artefact. The 3-D recording of the samples should make it possible to correct for this, by comparing elemental composition with height.

4.4.2: Methodology

ed Plasma Emission Spectrometry was the method chosen. It involves the dissolution of a known quantity of sample by dilution to a fixed concentration in distilled water. The sample is then introduced to the plasma flame, and the emission position measured by a polychromator, which measures the wavelength and intensity of the emitted radiation. It is a technique that has been largely developed for geological analyses, of rock samples.

Sample preparation methods developed for specific materials are used. The one chosen for our purposes was a powerful hydrofluoric acid digestion of the ground samples.

The technique is the ICP torch, which is a two (or more) stage torch. A high frequency current. When the current flows, a magnetic field is generated within the coil. Charged particles pass through the magnetic field cut the force lines and are deflected. The sample is introduced into the coil field as an aerosol. The sample is introduced into the coil field as an aerosol gas nebuliser, and 'ignited' with a Tesla coil. The plasma flame generated is at a temperature of $\sim 10,000^\circ\text{K}$ that no chemical bond can survive, and so the analyte can be considered to be complete. The sample is introduced into the outer tube, and the sample is introduced to the cooler centre of the flame. As a result, heating of the sample from the outside inwards, thus minimising self-adsorption effects, and leading to calibration lines that are linear over five orders of magnitude. The extreme temperature generates strong emission lines which are detected by the

major methods of operation of the spectrometer, are the sequential and the simultaneous systems. The sequential system uses a monochromator which can be rotated under computer control over a wide range of spectral lines, and measurement by a series of detectors, one for each wavelength. The simultaneous counting system uses a polychromator to measure several wavelengths and the same time. The latter method limits the number of elements to about thirty, but enables much quicker throughput.

Sample preparation is the grinding of the samples to a fine consistency. This improves the efficiency of the dissolution process by increasing the available surface area of the material, and enhances the rate of dissolution. The material is then digested in an aqua regia mixture, to break down the bonds in the sample. The HF/HClO₄ is then driven off by heating, and the sample dissolved in concentrated hydrochloric acid. After heating the sample is made up to a known concentration with distilled water and is then being introduced into the plasma flame.

The system used comprised a Philips PV8490 ICP source unit, with a detection system in operation. The detection system was capable of 25 elements.

4.4.3: Results of the ICP analysis; Introduction.

There follows a description of the results from ICP analyses carried out by Dr.Hart at QMC, and by J.Miles for the Leverhulme Project. It would be impossible to present all the results from the larger analysis in the given space, so it has been decided to give here a fuller account of the external work, in order to illustrate the value of the technique. A fuller version of the 'in-house' experimental results will be published elsewhere. The same array of elements was examined in both experiments.

4.4.4: ICP results from QMC.

The number of samples submitted to, and analysed by Dr.Hart, do not provide a statistically viable range, for quantitative purposes. However, as an introduction to using this technique for bone and soil analyses, the results provide some very useful qualitative pointers towards the sort of trends to be expected in a wider scale sample series. One of the reasons for running these samples was to get some immediate idea of the effect, if any, of the Vinamul consolidant spray on the elemental/trace elemental content of the material from the burials, and as mentioned above, to see if the older samples had been affected by their storage. (The details of the experimental procedure are given elsewhere. The work was carried out at Royal Holloway and Bedford New College, Egham, Surrey, on the same machine as our later analyses)

The trends discussed here refer to the table of results below. Bovine bone was used for the fresh bone comparison, due to the difficulties encountered in obtaining fresh human bone. No statistical treatment of these figures has been undertaken, the observations are simply empirical.

Referring to Table 1 below, it is apparent that the unaltered grave fill, as represented by the first four samples, is very homogeneous, with an extremely regular elemental make-up. The iron/aluminium ratio is between 3 and 4:1, and there are minimal contents of calcium and phosphorous (phosphate), which are the main constituents of the mineral phase of bone. The actual bone samples are clearly characterised by massive peaks at the calcium and phosphorous readings, with relatively low percentages of other elements. The trace-element content is also distinctive, with enhanced amounts of copper, niobium, strontium and zinc, and very clear dearths of material represented in the other samples. The proportions of elements in the fresh bovine bone and the archaeological specimens are quite different, although the peaks are in the same places. It is not certain whether this is a result of intrinsic differences in the bones of two different species, or whether it reflects the diagenetic effects on the buried bone. The real interest lies in the group of soil samples between the grave fill and the bone columns, clearly different from the background provided by the fill.

The points that this set of samples have in common, by comparison with the grave fill levels, are:

- a) some alteration in the iron/aluminium ratio, in some cases a clear enhancement of aluminium, and generally lower iron levels;
- b) a clear enhancement in the levels of calcium and phosphorous;
- c) a general increase in the contents of the following trace elements; barium, cerium, cobalt, chromium, lanthanum(?), strontium.

Discussion.

The critical diagnostic elements for differentiating the 'body stain' samples from the grave fill appear to be calcium and phosphorous. The enhancement of these elements over the background level must be derived from the body tissues, almost certainly from the bone itself. This is clear for all five central samples in Table 1 (E,J,K,F,B). However, the two samples with the lowest calcium and phosphorous readings, J and K, show far less obvious variations in the concentrations of the trace elements. In fact the trace element contents alone would not differentiate them from the grave fill samples. Neither of these samples was recognisable as 'body stain', but their elemental content clearly distinguishes them from the other visually similar grave fill samples. It is provisionally possible to suggest that the calcium and phosphorous contents of samples of unknown origin could be used to determine whether or not they were derived from a decayed burial, and that the presence of enhanced quantities of these two elements would be sufficient evidence of a burial where no visible traces remained (at least under the conditions prevailing at Sutton Hoo).

The three soil samples with more marked calcium and phosphorous contents, must be assumed to be closer to the centre of decay, and thus more affected by the decay products. Samples E, F, and B all have enhanced levels of calcium and phosphorous, but they also have an increased level of aluminium, and a relatively lower concentration of iron, (the aluminium/iron ratio is altered). The mechanism of this alteration is uncertain, but the conclusion is that the presence of the body decay products fixes aluminium as a replacement for iron. In other conditions, at Mucking, Essex, (Keeley et al. 1977), a strong manganese (Mn) enhancement was observed in the body silhouette, over the background level. It was inferred that the manganese was drawn in from the surrounding soil. There are unfortunately not enough properly located samples in this series to determine whether that is the case with the aluminium enhancement observed here, but it seems likely that some interchange with the surrounding soil has taken place. Incidentally, a comparable manganese enhancement is not apparent here - it has been suggested that the enhancement of one element rather than another is a pH dependent phenomenon.

The three clear body samples show the highest values of several of the trace elements, including a number not present in the bone. The inference is either that the enhancement comes from an interaction with the surrounding soil, as mentioned above, or that the 'extra' trace elements are derived from the soft tissues of the body rather than the bones. The presence of all the enhanced trace elements in the grave fill soil suggests that the increased quantities are derived from the soil - one might expect a different range of elements to be present in each set of samples, if the sources of those elements were different.

Another possibility is that the trace elements are derived from material added to the topsoil (ie. fertilisers, rain), and that during the leaching process, those elements are differentially adsorbed by the in situ body decay products.

One final note from the table of figures is that the pH readings from this set of samples are much higher than those previously taken at Sutton Hoo, (Dimbleby 1975), which were considerably more acidic (as low as 3.80). This may also be an effect of chemical additions to the topsoil.

The question of contamination derived from the use of consolidant sprays on the site must also be considered. The consolidant used is Vinamul, a water borne PVA emulsion. This is clearly an organic hydrocarbon, the basic elements of which cannot be measured by this method, as all organic material is destroyed during the digestion process. So the only real danger as regards the ICP analysis, is possible contamination by trace elements in the consolidant. The two clearly consolidated samples, E and J, do not show any trace element pattern distinctive from the other samples. They instead show a trace element distribution pattern related to their proximity to the burial, and do not appear anomalous in any other way - ie. J is closely related to K, and E clearly belongs with F and B. The two possible exceptions to this are the chromium and nickel contents of sample E; but as this is not reflected in the contents of those elements in J, it does not seem likely that they are consolidant derived.

Conclusions.

The main conclusion to be drawn from what must still be regarded as a very inadequate sample is that there are chemical differences between the areas affected by the decay products of the buried bodies, and the grave fills. It can be further noted that certain elements are diagnostic of this chemical difference, and as such provide criteria for chemical separation of body/non-body samples where visual differentiation is not possible. It also appears that the use of Vinamul consolidant on the grave material does not affect the elemental or trace elemental concentrations in that material, although it may affect the organic content.

References:

- Dimbleby, G.W. 1975 The environment, in Bruce-Mitford, R.L.S. (ed) The Sutton Hoo Ship Burial Volume I. British Museum, London.
- Keeley, H.C.M., Hudson, G.E. and Evans, J. 1977. Trace Element Contents of Human Bones in Various States of Preservation. Journal of Archaeological Science 4, 19-24.

Table 1: QMC ICP spectrometry results.

July 1986.

Major Elements % ↓	Sample +													
	A	C	G	H	E	J	K	F	B	D	I	L	M	
Al (Aluminium)	0.51	0.74	0.58	0.54	1.33	0.28	0.42	0.92	2.52	0.21	0.29	0.06	0.00	
Fe (Iron)	1.96	2.05	2.20	2.90	1.49	1.05	1.91	1.72	1.08	0.06	0.36	0.05	0.00	
Mg (Magnesium)	0.05	0.06	0.05	0.37	0.06	0.04	0.06	0.06	0.06	0.16	0.18	0.57	0.10	
Ca (Calcium)	0.33	0.72	0.42	0.06	2.57	1.71	2.54	4.70	3.78	44.59	45.01	36.42	0.10	
Na (Sodium)	0.06	0.13	0.08	0.03	0.08	0.08	0.08	0.17	0.18	0.43	0.46	0.76	0.00	
K (Potassium)	0.04	0.04	0.03	0.01	0.06	0.04	0.03	0.04	0.03	0.01	0.00	0.04	0.00	
Ti (Titanium)	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.04	0.04	0.01	0.00	
P (Phosphorous)	0.13	0.40	0.17	0.18	1.91	1.01	1.53	2.94	2.63	29.73	30.32	18.87	0.00	
Mn (Manganese)	0.02	0.02	0.02	0.02	0.03	0.02	0.03	0.04	0.02	0.00	0.02	0.00	0.00	

Trace Elements ppm, ↓

Ba (Barium)	20	36	20	23	144	37	59	95	116	74	29	29	3
Ce (Cerium)	10	13	7	4	48	2	6	28	53	-	-	-	-
Co (Cobalt)	5	9	5	6	24	8	10	31	19	-	-	-	-
Cr (Chromium)	6	7	7	17	152	19	20	28	30	-	-	-	7
Cu (Copper)	12	11	9	9	14	11	14	18	16	45	37	36	4
La (Lanthanum)	6	7	4	4	14	3	5	11	21	-	-	-	-
Li (Lithium)	2	2	1	1	3	1	2	4	4	9	8	2	-
Mo (Molybdenum)	3	3	4	3	4	4	4	4	2	-	-	-	3
Nb (Niobium)	2	2	2	2	4	2	2	5	7	28	28	3	-
Ni (Nickel)	20	16	15	22	83	11	15	17	16	-	-	-	4
Sc (Scandium)	1	1	1	1	3	-	1	2	6	1	1	1	-
Sr (Strontium)	17	25	18	18	42	27	40	48	93	212	228	74	6
V (Vanadium)	16	16	17	21	12	8	13	17	8	8	10	4	-
Y (Yttrium)	4	4	3	3	8	1	2	7	15	2	2	1	-
Zn (Zinc)	60	57	38	45	72	52	67	120	71	353	325	97	15
Zr (Zirconium)	-	1	-	-	1	-	-	1	3	2	5	1	-

pH (H₂O) 6.65 6.80 6.60Sample Key:

A = Find 2026, light soil concretion; flesh stain?
 B = Find 2026, dark soil concretion; body matrix.
 C = Find 2026, light soil; grave fill.
 D = Find 1985, bone.
 E = Find 1985, flat concretion; consolidant contamination.
 F = Find 1985, soil; grave fill.
 G = Find 2021, soil concretion; grave fill?
 H = Find 2021, soil; grave fill.
 I = Find 1994, bone.
 J = Find 1994, flat concretion; consolidant contamination.
 K = Find 1994, soil; grave fill.
 L = Fresh bovine bone.
 M = Blank.

Samples consisted of 0.25g of finely ground material.

4.4.5: Results of the in-house ICP analysis.

This work was carried out by J.Miles during 01/12/86 - 14/12/86, at the Geological Sciences Laboratory of the Royal Holloway and Bedford New College, (University of London), Egham, Surrey. The laboratory provided all equipment, and also the standard solutions. All corrections were made using computer programs already in use by the laboratory. We would like to acknowledge with gratitude the assistance given by Dr. Nick Walsh and the staff of the Geological Sciences Lab. at Royal Holloway.

The samples analysed consisted of 131 grave fill samples, 28 body samples, and 6 wood samples from the grave. Also analysed were 84 samples from the four columns in Intervention 43. These totals do not include 3 fill samples and 4 wood samples for which the computer adjusted results have clearly been wrongly calculated (eg. negative concentrations of elements appear). the results were received as tables showing the content of the following elements as % by weight of oxide: Aluminium (Al), Iron (Fe), Magnesium (Mg), Calcium (Ca), Sodium (Na), Potassium (K), and Manganese (Mn); and as parts per million: Barium (Ba), Cerium (Ce), Cobalt (Co), Chromium (Cr), Copper (Cu), Lanthanum (La), Lithium (Li), Molybdenum (Mo), Niobium (Nb), Nickel (Ni), Scandium (Sc), Strontium (Sr), Vanadium (V), Yttrium (Y), Zinc (Zn), and Zirconium (Zr).

It must be noted that this is essentially a trace element analysis, and does not include any organic material, or the bulk of the sand which is composed of silicates. We are still in the process of interpreting the results of this analysis, and so far have concentrated on the grave fill and body samples.

The statistical treatment of the results should have the following objectives:

- i) assess the reliability of the results;
- ii) outline any relationships between variations in elemental composition and location of sample within the same population;
- iii) outline any relationships between variations in elemental composition and the presence of grave artefacts;
- iv) Outline any relationships between variations in elemental composition with respect to that of other elements.

Point i) appears to be shown by the statistical analyses, ie. the results can be shown to be reliable, by the relatively normal distributions exhibited for the elemental concentrations when plotted.

ii): Simple graphical analysis of plots of the sample 'columns' reveal a clear homogeneity in the grave fill samples which it was felt would not be made any more clear through statistical analysis.

iii): The results of significance tests for each element, which give the probability of the concentrations within the two populations being distinguishable, are given in the table below.

Table 2: Ordered table of tests of significance:

Element:	Probability that the two populations have the same elemental compositions:	
Aluminium - Al	0.0000	
Cerium - Ce	0.0000	
Cobalt - Co	0.0000	
Lanthanum - La	0.0000	
Molybdenum- Mo	0.0000	
Phosphorous-P	0.0000	Group 1.
Potassium - K	0.0000	
Scandium - Sc	0.0000	
Titanium - Ti	0.0000	
Yttrium - Y	0.0000	
Calcium - Ca	0.0003	
Sodium - Na	0.0006	
Copper - Cu	0.0012	
Chromium - Cr	0.0014	Group 2.
Barium - Ba	0.0140	
Magnesium - Mg	0.0210	
Strontium - Sr	0.0260	
Niobium - Nb	0.0630	
Zinc - Zn	0.1200	
Vanadium - V	0.1500	Group 3.
Nickel - Ni	0.4000	
Iron - Fe	0.5800	
Zirconium - Zr	0.6900	
Lithium - Li	0.9700	Group 4.
Manganese - Mn	0.9800	

The elements in Group 1 show the clearest distinction between the two populations, and would therefore be the most useful indicators of the nature of a sample of uncertain origin. Group 2 has probability values within the 5% cut-off margin (0.0500), and so should also be reliable as distinguishing elements. Those in Group 3 represent overlapping of the two populations, but the analysis of more body samples might produce a more conclusive result for these elements. Group 4 elements are those for which the concentrations are indistinguishable between the two populations.

Point (iv) above, the relationship between the variations in elemental composition with respect to those of other elements, is currently being studied through the use of Stepwise Discriminant Analysis. This will show whether the concentration of any particular element is dependent on that of any other(s).

4.4.6: Discussion.

This analysis has shown that there is a distinct chemical difference between the material distinguishable as body silhouette or stain, and the background fill in which it lies. This is only to be expected, since the two can be distinguished visually. However, it is clear that an unknown sample could be labelled as body residue, from the pattern of elemental distribution revealed by ICP analysis.

The conclusions reached from the first set of results provided by QMC, can be extended and amended by reference to these more comprehensive figures. As seen from the earlier sample, calcium and phosphorous are shown to be definitely diagnostic as 'body' elements. The observed change in the iron/aluminium ratio is not statistically significant, as the iron content is not really distinguishable between the two populations. However, it can clearly be seen that aluminium is diagnostic. It is also present in the largest absolute quantities of the elements measured, so it may well be the best element to produce an indicator for. The presence of trace elements in a few parts per million, despite the clear enhancement shown over the fill samples, could be very difficult to show up by simple chemical means in the ground. We must, however, continue to study the bonding mechanisms of the various elements, as it may prove that elements present in larger quantities are not necessarily easier to release and react with a suitable indicator.

It can also be noted that the storage of the earlier samples, which it was felt might have affected the trace-element content, does not appear to have caused any clear changes, judging from the comparison with the later series.

The next stage of this work will be to compare the elemental composition of the grave material with that of the background soil, as sampled in the four columns of Intervention 43. The full statistical analysis has yet to be carried out, but preliminary graphical examination suggests that there is an enhancement of certain of the elements in the topsoil, eg. aluminium, calcium, phosphorous, sodium, titanium, copper, molybdenum, and others of the minor trace elements. What we do not know is whether the source of these elements is from some anthropic activity, such as fertilizer application, or exactly how they are bonded in the topsoil. It can be assumed that the bonding mechanism is similar to that operating in the body silhouette, which is probably some form of complexation with the humic material. It is not certain, however, whether the elemental enhancement in the body derives from capturing of material leached down from the topsoil, or directly from the body decay. This is vital to understand, if the findings from Sutton Hoo are to be relevant to any other site: for example, if the source of the body stain differences lies in the topsoil make-up, it would require a similar topsoil on another site to produce the same effect.

This exciting work is continuing, and it is hoped will provide some answers to the many intriguing questions raised by our initial investigations. Together with the results of the various organic analyses, we should be able to build up a comprehensive chemical portrait of the degraded burials at Sutton Hoo, and develop a tool for solving any future 'was there a body?' controversy.