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SUTTON HOO RESEARCH TRUST

Leverhulme Trust Project

Interim Report No.2 March 1988

P.Bethell (ed.)

Sutton Hoo Research Trust - Leverhulme Trust Project

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Sutton Hoo - Leverhulme Trust Project

Second Interim Report: February 1988.

Introduction.

The last year has been one of great interest in the project. The broad potential of the field of study has become even more apparent, although of course we have only been able to concentrate on certain aspects. The laboratory work has continued on the humic material from the Sutton Hoo burials, particularly on the relationship of the original material to the decay products encountered at present. Further investigation into the best method of extracting the humic material has also been undertaken, and the first steps taken towards developing a dye for use in the field.

The project has continued to be dogged by problems with the staff. Joanne Miles left after a year in the Soil Chemistry lab., in order to get married. We subsequently appointed Miss Lorraine Stewart as research student. There were further problems in this, as Miss Stewart was classified as an overseas student (despite being born in this country, and having British citizenship), and consequently we were required to pay higher fees for her. This situation is of course not fully satisfactory. The lack of continuity on the chemical side of the project has resulted in a shortfall in progress over expectations - however, the work done by Miss Miles has produced some very fascinating information on the origins of the humic material in the Sutton Hoo graves. Miss Stewart has in part been able to draw on the experience gained by Miss Miles, so has not had to start completely 'cold'. Her work on the humic extractions is detailed below.

Field work in the past year has of course included on-site work at Sutton Hoo. The progress into the second ship-bearing mound continued, with the discovery of a burial chamber beneath the mound being the high spot of the season. Several more flat-graves were investigated, and a revised sampling procedure used, which was both quicker and provided a more coherent array of samples for analysis.

Material has also been accessed from other sites, including Brandon, Suffolk; Atlantic Trading Estate, Barri, West Glamorgan, (both visited in person); Snape, Suffolk; and Burrow Hill, Suffolk. A good collection of comparative material has been built up.

It was hoped to continue the trace-element analysis, using the ICP facility at Royal Holloway College, Egham. An application was made by this body for funds from the SERC, to support ICP analysis of material from several archaeological projects. This would have provided 'free' analysis of a large number of samples. However, the application was eventually rejected, and this facility did not come into being. Apart from the disappointment of this failure, of course our own trace-element programme was put in abeyance while awaiting the outcome of the SERC application. Thus no more of this particular analysis has been

undertaken in the last year, where it was hoped to dramatically increase throughput of samples.

Collaboration with other institutions has proved fruitful in many ways, however. Workers at Cardiff University have examined the fatty acid content of samples from Sutton Hoo, as part of their work on material from other sites. Details of this are below. I have also explored the possibility of joint publication of certain aspects of our work with personnel at Cardiff.

My own work has continued to concentrate on the collation of information relating to other Anglo-Saxon cemeteries and the various states of preservation encountered on them. This is continuing, but cannot as yet be put into coherent form.

I also attended several conferences and day-schools during the year, the most fruitful of which was the Archaeological Science conference in Glasgow on 23rd-26th September 1987. A great many useful contacts, and much information exchange, were made. I am currently preparing a paper for the forthcoming Burial Archaeology conference in Bradford, on aspects of the Leverhulme Project work.

Publications over the last year have been:

Bethell, P. H. and Carver, M. O. H. (1987)

"Detection and enhancement of decayed inhumations at Sutton Hoo.", in Boddington, A., Garland, N. and Janaway, R. (eds.) Death, Decay and Reconstruction 10-21. (Manchester University Press, Manchester)

Bethell, P. H. and Máté, I. (In press)

"The use of soil phosphate analysis in archaeology: a critique." In Henderson, J. (ed.) Scientific Analysis in Archaeology, and its interpretation. (Oxford University Committee for Archaeology, Monograph 19; Oxford)

Bethell, P. H. and Smith, J. U. (In preparation)

"Trace element analysis of an inhumation from Sutton Hoo, using inductively-coupled plasma emission spectrometry (ICP): an evaluation of the technique applied to analysis of organic residues."

Philip Bethell,
University of Birmingham,
Feb. 1988.

Report on organic analyses: Joanne Smith (née Miles).

A number of organic analyses have been undertaken in the past year, including preliminary amino-acid, polysaccharide, and HNO analyses. Some results of interest were obtained. For example, there was a clear distinction between the amino-acid content of the wood and the body residues, especially in the amount of cystine, which was much higher in the wood sample. This could lead to a method of distinction between such residues. The results of this and other analyses are currently being processed as part of a larger report, the structure of which will be roughly as follows:

- 1) Background on organic material in soil.
- 2) Derivation of equation to interpret results.
- 3) Brief background of amino-acid analysis.
- 4) Use of equation to analyse amino-acid results.
- 5) Brief background of carbohydrate analysis.
- 6) Use of equation to analyse carbohydrate results.
- 7) Report of results obtained for humic acids, and humic acid background.

This is being written as part of an MSc report, and is not yet finished, as Mrs Smith has begun another project at Reading University. However, these results will be presented as soon as they are put into coherent form.

Preliminary analysis of Sutton Hoo material for fatty acid traces.

Work undertaken by G. Davies, Department of Chemistry, University College Cardiff.

"The residues obtained from the Sutton Hoo soils were subjected to infra-red spectroscopy. These only showed the presence of C-H stretches (ca. 3000 cm^{-1}), except the lower leg which showed O-H (c. 3400 cm^{-1}), C=O (1745 cm^{-1}) and C-O (c. 1150 cm^{-1}) (carboxylic acid?).

Thin layer chromatography was performed on the residues. Using hexane/ether as the developer there was no evidence of sterol presence. However, using a more polar developing solvent system ie. ether, ethyl acetate, ethanoic acid and petroleum ether, there was evidence of fatty acid presence ($R_f \approx 0.6$). This evidence was not present in the background samples. In all the Sutton Hoo residues there was evidence of hydrocarbon presence from TLC, (ie. components at the solvent front $R_f = 1$, which means that this is non-polar and non-retained, and possibly hydrocarbon).

NMR spectroscopy of sample 3321 (Head) showed no olefinic protons but extensive overlap of signal in the range δ 0.4 - 2.2. No gas chromatography - mass spectrometry (GCMS) was undertaken on these samples, but would obviously need to be done before any firm statements can be made concerning the precise constituents of the residues."

This brief report shows that there are traces of fatty acids in the body samples, and not in the background, suggesting that these are derived from the body decay products. Further work in this area could show the exact nature, and thus the derivation of the fatty acid traces. The Cardiff report also contains information on work carried out on samples from a body on the site at Atlantic Trading Estate, Barry, W. Glamorgan.

Sutton Hoo - Leverhulme Trust Project

Laboratory work: Lorraine Stewart.

This project has two main aims. The first is to determine the origin(s) and retention mechanism of the organic matter in the body silhouette. The second is to establish enough difference between the body and grave fill regions based on either the organic or inorganic soil constituents, and hence to develop a dye to show up the outline of a body. In order to investigate the properties, characteristics etc., of the organic matter, the humic substances need to be extracted and analysed.

The inorganic content of the soil sample has been determined by Inductively-Coupled Plasma Emission Spectrometry (ICP). This is a fast, destructive method of determining the amounts of trace elements present, but does not give results on organic matter or silicates, which are the major part of sand.

Since joining the project last August, I have been concentrating on examining the humic material, and have been exploring methods of extraction of humics from the soil, along with attempts to refine the process of humic material reduction using sodium amalgam degradation.

Humic Substances.

Humic substances are the most abundant organic macromolecular organic substances. In the soil samples collected at Sutton Hoo they were brown in colour. They are the major contributors to soil aggregation by virtue of their stabilizing effect on soil aggregates. It is hoped to ascertain the origin(s) of the humic substances in Sutton Hoo soil samples by their chemical composition. The humic substances could have been derived from:

- 1) Lignins; the lignins (found abundantly in wood) are believed to undergo peripheral oxidation thereby producing humic substances.
- 2) Chemical Synthesis; there is random combination of amino-acids, proteins and other organic molecules.
- 3) The humic substances in the grave fill samples could also be derived from the body by virtue of decomposition.

Humic substances are divided into three main groups. Those which are soluble in both acid and alkali are called Fulvic Acids, and these tend to be the most easily oxidised and are the lowest molecular weight humic substances. Those which are soluble in alkali, but insoluble in acid are classified as Humic Acids, and these are usually very dark in colour. The Humins are those which are soluble in neither acid or alkali, and hence not much study has been carried out on this fraction. (See figure for the classification). Humic substances are known to be macromolecules consisting of free phenolic and carboxylic groups which renders them anionic species.

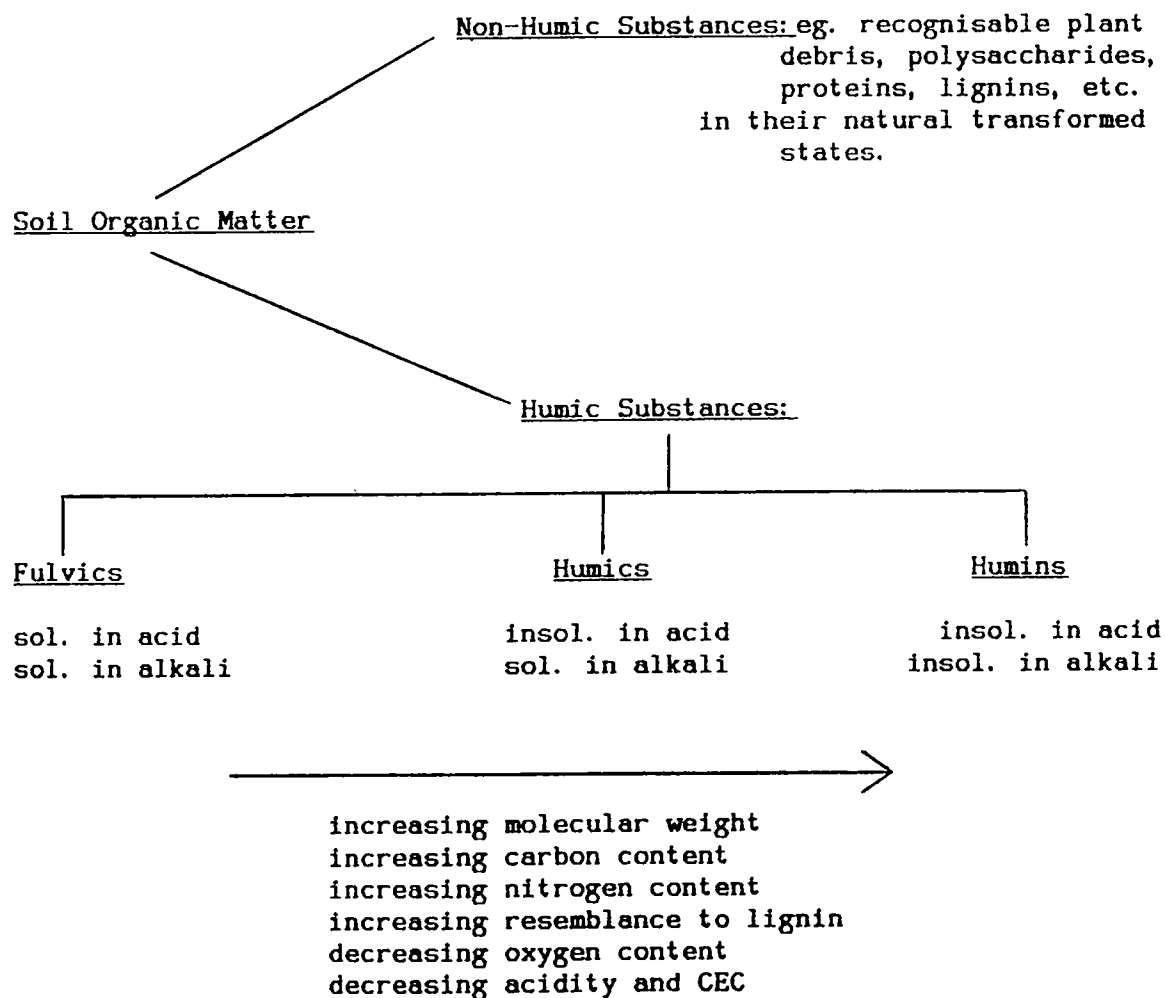


Fig. 1: Schematic diagram of soil humic fractions.

Extraction and Fractionation of Humic Substances.

The extraction of humic substances from soil samples collected from the Sutton Hoo sites was investigated using sodium hydroxide, sodium pyrophosphate, and 6% dimethylsulphoxide (DMSO)/conc. hydrochloric acid mixture as extractants. The ideal extractant should remove practically all the humic substances from the soil without altering physical or chemical properties. However, one has to bear in mind the quantitative aspects of the extraction procedure when dealing with small quantities of sample if complete characterisation is to be carried out. Hence when choosing the most effective extractant both points were considered.

The criteria for effective solvents for extraction of humic substances are:

- a) high polarity and a high dielectric constant to assist the dispersion of charged molecules.
- b) a small molecular size to penetrate the humic polymer.
- c) the ability to disrupt the existing hydrogen bonds and provide alternative groups to form humic-solvent hydrogen bonds.
- d) the ability to immobilize metallic cations.

So far, the most effective solvent on a quantitative basis has been dilute sodium hydroxide. However, each solvent has its advantages and disadvantages which will be discussed.

Sodium hydroxide is a good solvent for the extraction of humic substances. It has proved itself to be most effective (quantitatively) in the extraction of the higher molecular weight humic substances, i.e. humic acids. The primary disadvantages of sodium hydroxide as an extractant is that under alkaline conditions auto-oxidation of humic substances tend to occur. However, this is inhibited by carrying out the reaction (extraction) under nitrogen to create an inert atmosphere. The soil also has to be pretreated with 0.1M hydrochloric acid for decalcification purposes. Decalcification is necessary to break the mono- and poly-valent cation-humic substances bonds, so dissolution of the humic material can occur. The acid dissolves the metal ions, hydrous oxides and hydrated silicate minerals associated with the humic substances. It also dissolves the lower molecular weight fulvic acids so this extract must be kept and combined with the other sodium hydroxide extracts. Hence because the disadvantages can be compensated for, it was decided to use sodium hydroxide as the extracting solvent.

Dimethyl sulphoxide is a dipolar aprotic organic solvent. It removes large amounts of the lower molecular weight (fulvic acid) material under non-oxidative conditions. It is not very efficient in removing the higher molecular weight humic acid from the sandy mineral soil collected from Sutton Hoo. It is not necessary to pretreat the soil with hydrofluoric acid although DMSO is a poor solvent for anions. The addition of 1ml of conc. hydrochloric acid to 99ml of DMSO (1% conc. HCl/DMSO mixture) is enough for efficient extraction of humic substances from peat soils because the H^+ humic substances are produced. However, with sandy mineral soils, where the concentration of humic substances is low and that of the mono- and poly-valent anions is high, a 6% HCl/DMSO mixture was needed for efficient extraction. The DMSO needs to be

thoroughly removed from the humics before starting the fractionation by the use of 0.1M HCl. The humic acid and fulvic acid extracted with DMSO/HCl tended to be much darker than that extracted with sodium hydroxide. The reason for this has not been ascertained.

Sodium pyrophosphate is a very mild extractant. It is an aqueous salt solution neutralized to pH 7.0 with 6M HCl. The soil does not need to be decalcified as in the case of NaOH because the pyrophosphate ($P_2O_7^{4-}$) is a very good chelating agent. The pyrophosphate anion chelates the calcium cation forming calcium pyrophosphate (CaP_2O_7) and the sodium salt of the acid is formed, hence dissolution of the humic material occurs. The reaction may be represented as follows:
 $Na_4P_2O_7 + Ca^{2+} - \text{humic-COO}^- \rightarrow Ca_2P_2O_7 + \text{humic-COONa}$

The problem encountered with the sodium pyrophosphate is that it solubilizes only the more polar compounds hence the phenols which do not ionize to give the phenolate species will not be extracted. Sodium pyrophosphate extracts mainly the humics containing carboxylic groups but not those containing phenolic groups. Hence the merit of this solvent was fairly limited for the objective of this project due to the fact that it is very selective and quantitatively inefficient. This solvent would be useful when in-depth studies of the classes or groups of humic acid and fulvic acid is pursued.

Procedure for the Extraction and Fractionation of Humic Substances.

The procedure for the extraction and purification of humic and fulvic acid from the soil samples is almost the same for all three solvents, hence a general method will be outlined.

Preparation of solvent:

a) DMSO/HCl - A 6% solution of HCl in DMSO was prepared by adding 6ml of conc. HCl to 94ml DMSO. NB: the drier the DMSO the more efficient the extraction.

b) Sodium hydroxide - 1 litre of 0.1M NaOH was prepared under nitrogen by dissolving 4g NaOH pellets in some distilled water, and then making up to the litre mark in a volumetric flask.

c) Sodium pyrophosphate - 1 litre of 0.1M $Na_4P_2O_7$ was prepared by dissolving 44.6025g of $Na_4P_2O_7$ crystals in some distilled water in a beaker. The volume was adjusted with water to about 950ml, and then the pH of the solution was adjusted to 7.0 using 6M HCl. This mixture was transferred to a 1 litre volumetric flask, and made up to the 1 litre mark with distilled water.

Extraction of humics using NaOH:

- 1) To 10g of soil add 100ml 0.1M HCl, in a plastic bottle.
- 2) shake the suspension for 12hrs on a miller roller.

- 3) separate the supernatant by centrifuging at 13k for 30mins.
- 4) wash the soil with distilled water and combine washings with supernatant from (3).
- 5) add 100 ml 0.1M NaOH to the soil under N_2
- 6) extract the suspension by shaking continuously for 12 hrs.
- 7) separate supernatant by centrifuging at 13k for 30 mins.
- 8) repeat steps 5,6, and 7 until extracts become clear.
- 9) combine all portions of extracts.
- 10) acidify the supernatant to pH 1 to precipitate the humic acid from solution leaving a solution of fulvic acid.
- 11) allow suspension to stand for 12hrs.
- 12) centrifuge for 30mins. at 13k and remove supernatant.

When the extraction was carried out using $Na_4P_2O_7$ and DMSO/HCl, steps 1 to 4 were eliminated, the other solvents being substituted at step (5).

Fractionation of Fulvic acids:

- 1) pass the supernatant through an XAD-8 column to remove impurities from the fulvic acid.
- 2) wash the unabsorbed amino-acids, polysaccharides, chloride ions etc. from the column with distilled water until chloride free (test for Cl^- with 0.1M $AgNO_3$).
- 3) when the effluent is chloride free, the column is back eluted with 0.1M sodium hydroxide solution.
- 4) the back eluted fulvic acids should be immediately passed down an H^+ -saturated cation exchange resin to remove exchangeable Na^+ ions because fulvic acids are easily oxidised at high pH.
- 5) freeze-dry the acidified effluent which contains H^+ exchanged fulvic acid to cause minimal structural alteration.

Fractionation of humic acids:

- 1) dissolve the residue in a minimum amount of 1% conc. HCl in DMSO, and treat in the same manner stated above for the fulvic acids. The DMSO is removed from the XAD-8 column by washing it through with 0.1M HCl before proceeding to step (2).

NB. XAD-8 is a hydrophobic macroporous resin which acts as a molecule sieve, trapping humics in its holes while allowing amino-acids etc. to pass through.

Using sodium pyrophosphate the fractionation of the fulvic and humic acids was the same as that outlined for sodium hydroxide. However, there is an additional step in the case of 6% HCl /DMSO. The DMSO was washed from the column after step (a) with 0.1M HCl . The humic substances were then back eluted from the column using 0.1M NaOH and treated in the manner stated above starting from the step 10 in the extraction. The results are tabulated below.

| Solvent | Wt. of product. | | mg. product/g soil sample | |
|-----------------------------------|-----------------|-------------|---------------------------|-------------|
| | Humic acid | Fulvic acid | Humic acid | Fulvic acid |
| NaOH | 54.6 | 13.6 | 3.64 | 0.91 |
| 6% HCl /DMSO | 20.0 | 20.6 | 1.33 | 1.31 |
| $\text{Na}_4\text{P}_2\text{O}_7$ | 32.0 | 25.3 | 0.64 | 1.01 |

Sample no. 20235 from Sutton Hoo.

Characterisation of the freeze dried H^+ -fulvic acid and H^+ -humic acid will be done by use of Infra-red Spectrometry (IR) which will reveal the functional groups present, microanalysis will tell the nitrogen, carbon and hydrogen content, and the oxygen can be calculated by the method of difference: macroanalysis will yield the amounts of various sugars and amino-acids present in the humic and fulvic acid fractions. The micro-analysis results will be used to reveal if and how much oxidation does take place when sodium hydroxide is used as the extracting solvent based on the oxygen content.

Sodium Amalgam Degradation of Humics.

So far the area of analysis of the products being investigated is that of sodium amalgam degradation. This is not a fully developed process, therefore a lot of reading work and experimentation has been done to investigate the process and perfect the procedure. The sodium amalgam degradation (reduction) is supposed to break ether linkages ($\text{C}-\text{O}-\text{C}$) of chains in the humic and fulvic acid molecules thereby producing phenols which need to be methylated to render them volatile enough for identification by gas chromatography/mass spectrometry. It is hoped that the methylated product of the sodium amalgam degraded humic acid will give an indication of the origin of the humic substances, especially that extracted from the body samples. We also hope to discover the mechanism by which these humic substances are held in the soil.

The degradation process consists firstly of making the 5% sodium amalgam and secondly reducing the humic substance as extensively as possible into identifiable fragments.

Preparation of 5% sodium amalgam:

- 1) weigh out 1.5g of clean sodium into a beaker containing dry toluene.
- 2) transfer the sodium in toluene into a round bottomed flask containing approximately 15ml dry toluene. Melt the sodium in an apparatus set up as below (Fig. 2), using a hot plate with a magnetic stirrer.
- 3) remove the heat and slowly add 30g of mercury dropwise. The reaction is vigorous and keeps the toluene boiling so most of it should have evaporated by the end of the reaction, but the vapour protects the amalgam from air. If excess toluene is left at the end of the reaction, decant off before the amalgam solidifies.
- 4) pour the amalgam into a mortar with a tightly fitted rubber cap, and pulverise using a pestle: note the amalgam is very susceptible to attack by air, therefore it must be used within a few minutes of preparation.

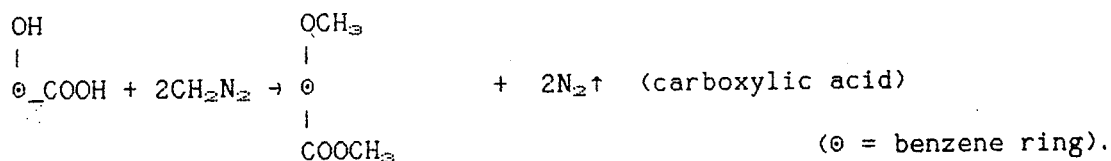
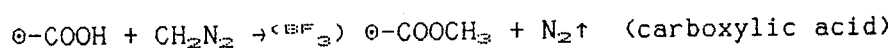
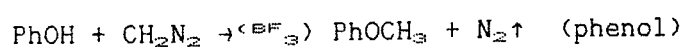
Sodium amalgam degradation:

- 1) pass nitrogen through the apparatus set up as in Fig. 2 for 30 mins. to displace air from the apparatus.
- 2) place 30g sodium amalgam in a 100ml round bottomed flask.
- 3) add 1ml methanol to reduce the tendency of the solution to foam at step (5).
- 4) dissolve 25mg humic acid in 15ml 0.5M NaOH soln.* and transfer to a dropping funnel.
- 5) slowly add the humic acid solution from the dropping funnel to the amalgam in the flask. Allow the initial vigour of the reaction to subside before heating the flask to 100-110° C for 3 hours.
- 6) acidify the reduced humic acid to pH 1.0 with 6M HCl.
- 7) decant supernatant solution from the mercury, extract with three 75ml portions of dry ether. NB: If an emulsion forms, open the top of the separating funnel, and remove some of the aqueous layer.
- 8) dry the ethereal solution of the product with anhydrous magnesium sulphate.
- 9) concentrate using rotary vacuum evaporator.
- 10) methylate the reduced humic acid.

The validity of the degradation cannot yet be fully assessed because a proper methylation method was only recently found, and it has not yet been applied to the reduced humic acids. The extraction with ether proved not to be very efficient, and therefore carbon tetrachloride was used to extract another portion of the reduced humics.

The progress made thus far is the discovery of a method for the methylation of the sodium amalgam degraded humics. The method of methylation used by Piper and Posner (1972), and M. Schnitzer (1974) which employed diazomethane proved completely unsuccessful with phenols and hydroxybenzoic acids, but it was 80-90% successful with carboxylic acids. It was discovered that the methylation process was not working when the results from the GC/MS analysis were obtained, which showed there were no ether compounds present. On discovering this the methylation process was tried out on a few standards, (phenols, carboxylic acids and hydroxybenzoic acids) to find the success or failure rate as stated above.

Since diazomethane did not methylate phenols and hydroxybenzoic acids by itself, the next step was to use it in conjunction with boron trifluoride as a catalyst. The BF_3 was used in the hope of lowering the activation energy of the methylation reaction enough to allow methylation to take place. The reactions are summarised as follows:

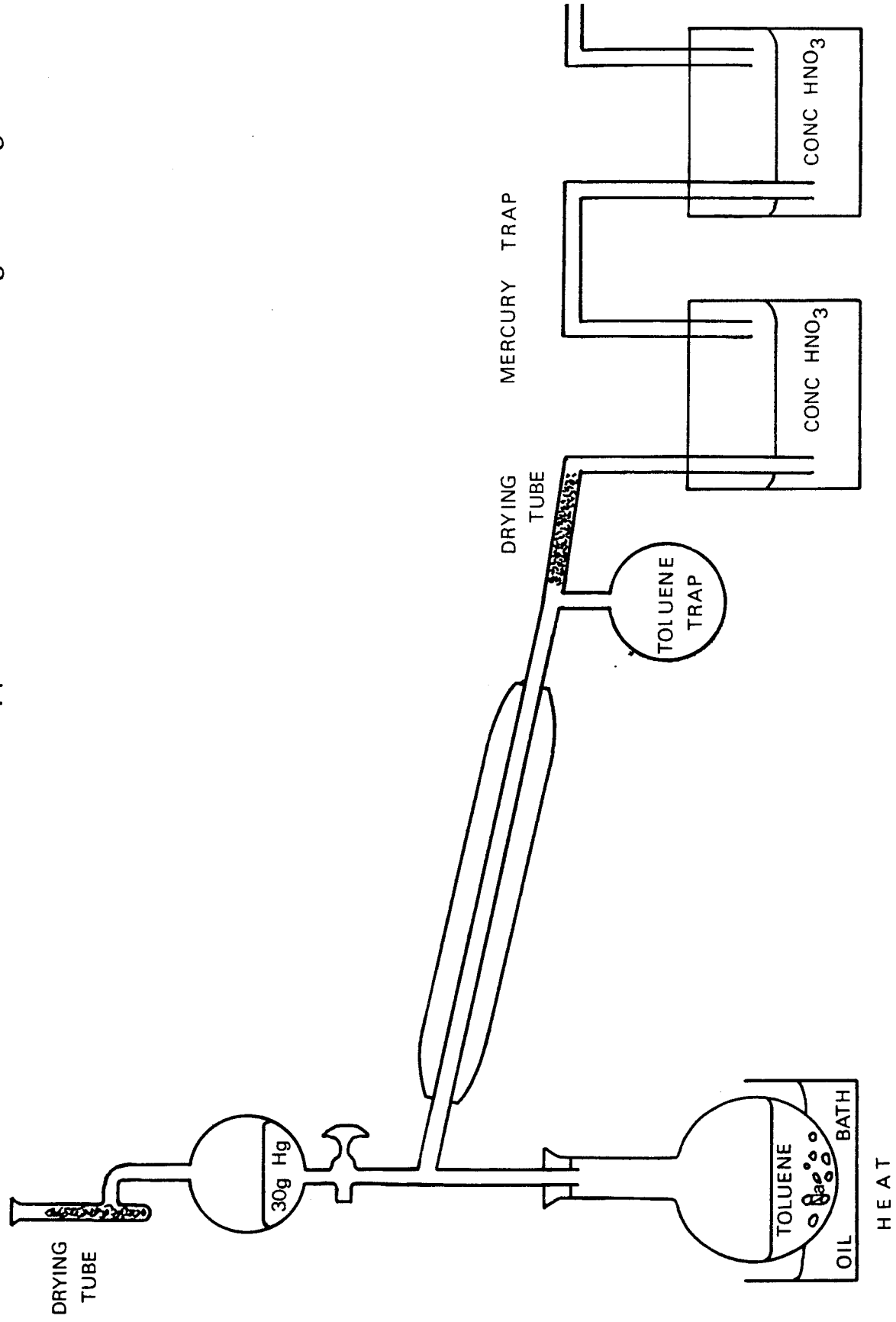


The boron trifluoride did not lower the activation energy enough in the case of phenols. Hence there was very little methylation in the case of phenols and hydroxybenzoic acids, which again was checked by the use of infra-red spectrometry. The CH_3 (methyl group) band occurs at a wavenumber of $2800\text{-}3000\text{ cm}^{-1}$ and you simply look for the band to determine whether or not methylation has occurred. There is also the disappearance of the OH and COOH bands which occur at 3000 cm^{-1} and 1700 cm^{-1} respectively.

The next method tried was one suggested by Dr. Neil Baggot in the Chem Dept., University of Birmingham, and this proved to be very successful. The reason this method, methylation using methyl iodide, was successful was because potassium dimethyl was used to produce the phenolate anion which can then be readily methylated. With phenols and mono-substituted phenols the phenolate anion has canonical forms which results in the production of two or three methylate products. However the phenols encountered in reduced humics are expected to be at least disubstituted thereby suppressing this problem. Hence a fairly good method of methylating reduced humic substances has been found.

A few modifications have been made to the method outlined by Piper and Posner (1972).; however, the optimum conditions for the degradation reaction have not yet been ascertained. Much effort is being channelled into the process as a complete successful breakthrough could prove invaluable to both soil chemists and archaeologists.

FIG. 2 Apparatus for sodium amalgam degradation



Refs.

Aiken, G., McKnight, Diane, Wershaw, R. and MacCarthy, P. (19xx)

Humic Substances in Soil, Sediment and Water John
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The Chemistry of Soil Processes Wiley and Sons,
Chichester.

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evaluation of the method. Soil Biological
Biochemistry

Schnitzer, M. and Khan, S. (1972)

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New York.

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The sodium amalgam reduction of soil and fungal
humic substances. Geoderma 9; 119-128.

Schnitzer, M. (1974)

The methylation of humic substances. Soil Science
117, 94-102

Swift, R.S. and Posner, A.M. (1972)

Auto-oxidation of humic acid under alkaline
conditions. Journal of Soil Science 23, 381-393.

Inorganic Analysis:

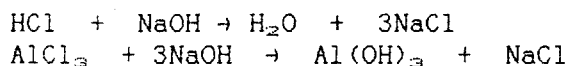
Miss Joanne Miles had originally started this project. She performed some ICP analysis on the samples collected from Sutton Hoo in 1986. Those results revealed a much higher concentration of Aluminium (Al) as the oxide in the body region samples than the gravefill samples and so it was decided to pursue some studies in this area. The aim of the investigation was to ascertain if there was a big enough difference in the concentration of Aluminium in the body and gravefill samples and hence to develop a dye which would show up the difference by staining (colouring) the outline of the body.

There are various types of aluminium present in the soil. The total aluminium comprises mainly the extractable and exchangeable aluminium. The exchangeable Al which is very closely related to the exchangeable hydrogen (H) is obtained by leaching the soil with a normal solution of a salt of a strong acid eg. potassium chloride (KCl) as was used in this analysis. The exchangeable H is also a measure of the acidity of the soil and in conjunction with the exchangeable Al it gives an indication of the weathering status of the soil. Exchangeable Al is adsorbed more strongly, and is active only under relatively acid conditions and is also thought to be adsorbed and desorbed from exchange sites like any other cation.

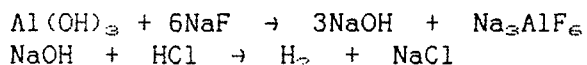
The extraction of Al is based on two requirements. Firstly, there is a high concentration of ions to displace the exchangeable Al and secondly, the solution into which the Al is displaced must maintain the Al in the soluble form. The use of KCl as the extracting solvent met with the first requirement; however, it was not in total agreement with the second requirement because the exchangeable Al in the soluble form exists only at a pH below 5.0. The soil from Sutton Hoo proved not to be as acidic as previous literature had described, and since the KCl is an unbuffered salt when it passes through the soil, it takes some time before the salt is at the same pH as the soil. During this period before equilibrium is attained, some of the Al is lost by precipitation from the extracting solution prior to removal from the soil. The method of extraction outlined in Black et al., (1965: 988) proved to be inefficient and unsuccessful. In this method 10g of the soil was placed in a 'fluted' filter paper in a filter funnel, and leached with 100ml of 1M KCl soln., using small aliquots at a time and taking no less than 2 hrs. The volume of the filtrate was then adjusted to 100ml with 1M KCl solution and the solution mixed thoroughly. However, some improvisation was necessary and instead of doing what was just outlined the soil and the KCl (as in same quantities as above) were mixed together for 3 hours on a hot plate with constant agitation using a magnetic stirrer.

The concentration of Al present in these solutions was determined by colourimetric measurement using Eriochrome Cyanine R. An attempt was made to determine the concentration of Al titrimetrically, but the concentration of Al was so small it could not be done. It was due to the use of titrimetry that it was realised that the soil was not very acidic. Since KCl (with negligible buffering properties) was used to replace the exchangeable Al and H from the soil, the leachate was

titrated with a standard base (NaOH) to obtain a measure of the total acidity (exchangeable Al and H). The solution was titrated to a pink end point using phenolphthalein as the indicator. The reactions involved on determining the total acidity by titration of the extracts with the base are summarised as follows:



Sodium fluoride was then added to the titrated solution to convert the Al in the Al(OH)_3 to a stable complex ion of fluoroaluminate, and sodium hydroxide is produced. The amount of Al is then ascertained by titrating the sodium hydroxide with standard hydrochloric acid. The reactions are summarised as follows:



The Al was present in small quantities ie. parts per million, as was indicated in the colourimetric method, hence the titrimetry was not a sensitive enough quantitative method.

Eriochrome Cyanine is brick-red in colour and yields a red-violet Al product in an acetate buffered medium (pH approx. 3.8-6.0). The method is quite simple but it is important to add the reagents in the exact order stated in the procedure. The Eriochrome Cyanine-aluminium complex forms rapidly in slightly acidic solutions but more slowly at pH 6. Hence the colour forming reagent must be added before the ammonium acetate buffer, otherwise low readings result from retarded colour development. The sample must be adjusted to pH 6 before the readings are recorded, because at pH values below 6 the Eriochrome reagent itself has a high absorbancy at $535\mu\text{m}$, which is the wavelength at which the absorbance of the samples are measured. At pH 6, the reagent background colour is minimal compared to the aluminium complex.

The only ion that interferes in the analysis for exchangeable Al is iron, and this is removed by complexation with sodium thioglycollate. The purple iron-thioglycollate complex begins to form at pH 6. The interference remains constant at concentrations of iron greater than $100\mu\text{g}$. Hence $200\mu\text{g}$ of soluble ferric iron was added to each sample so that the transmittance interference from the Fe-thioglycollate complex is relatively constant in all samples. The method is very simple, and consists solely of adding 2ml of the iron-compensating solution, 10ml sodium thioglycollate soln., 5ml Eriochrome Cyanine R soln., and then 10ml ammonium acetate soln. in that order. the solution was then diluted to a volume of 50ml and mixed thoroughly. After 18 minutes the light transmission at $535\mu\text{m}$ was read by use of UV spectrometer and the concentration of aluminium found by comparison with a standard curve.

The standard curve was prepared by making up a stock Al solution of known concentration and then making a range of standards and measuring their absorbance at the above wavelength of $535\mu\text{m}$. The log of absorbance was then plotted against the concentration of aluminium to

obtain the standard curve. By extrapolation the concentration of Al in the soil can be ascertained.

Results: Fill samples

| Sample. No. | wt. of sample (g) | Absorbance | mg Al/g of soil |
|-------------|-------------------|------------|-----------------|
| 3052 | 2.4965 | 0.216 | 0.421 |
| 3053 | 2.5005 | 0.163 | 0.270 |
| 3054 | 2.4995 | 0.100 | ---- |
| 3074 | 2.5000 | 0.110 | ---- |
| 3075 | 2.5012 | 1.041 | 1.164 |
| 3076 | 2.5006 | 0.130 | ---- |
| 3115 | 2.4992 | 1.280 | 1.050 |
| 3116 | 2.4999 | 0.607 | 0.935 |
| 3651 | 2.5001 | 1.195 | 1.274 |
| 3652 | 2.5009 | 1.240 | 1.299 |
| 3653 | 2.5007 | 1.010 | 1.189 |
| 3658 | 2.4990 | 0.920 | 1.146 |
| 3659 | 2.5004 | 1.230 | 1.289 |
| 3660 | 2.4990 | 0.872 | 1.116 |
| 3672 | 2.5012 | 0.108 | ---- |
| 3673 | 2.5006 | 0.255 | 0.535 |

Standard Curve Data:

| Volume of 5ppm stock soln. used/ml. | Absorbance | µg Al/50 ml soln. |
|----------------------------------------|------------|-------------------|
| 1 | 0.121 | 5 |
| 2 | 0.282 | 10 |
| 3 | 0.682 | 20 |
| 4 | 0.835 | 25 |
| 5 | 0.854 | 30 |

Wt. of $\text{Al}(\text{SO}_4)_3 \cdot 12\text{H}_2\text{O}$ = 0.4389g

A 25ppm stock soln. was made and from this a 5ppm stock soln.

The volume of 25ml leachate used for colourimetric analysis is 10ml.

Results: Body samples

| Sample. No. | wt. of sample (g) | Absorbance | mg Al/g of soil | |
|-------------|-------------------|------------|-----------------|---|
| 3551 | 2.5000 | 1.645 | 5.450 | |
| 3552 | 2.5004 | 1.599 | 5.048 | |
| 3553 | 2.5004 | 1.542 | 4.287 | S |
| 3554 | 2.4989 | 1.357 | 1.702 | E |
| 3555 | 2.4992 | 1.416 | 2.622 | T |
| 3556 | 2.5001 | 0.050 | ---- | |
| 3558 | 2.4997 | 1.300 | ---- | I |
| 3559 | 2.5005 | 0.700 | ---- | |

| | | | | |
|-------|--------|-------|-------|---|
| 3560 | 2.5007 | 1.469 | 3.749 | |
| 3563 | 2.5038 | 1.000 | 1.177 | |
| 3564 | 2.5034 | 0.432 | ---- | S |
| 3590 | 2.4992 | 1.005 | 1.180 | E |
| 3592 | 2.5007 | 1.596 | 5.297 | T |
| 3593 | 2.5001 | 1.239 | 1.290 | |
| 3595 | 2.5011 | 1.521 | 4.396 | I |
| 3600? | 2.5000 | 1.380 | 2.500 | I |

Standard Curve Data:

| Volume of 5ppm stock soln. used/ml. | Absorbance | $\mu\text{g Al/50 ml soln.}$ | |
|----------------------------------------|------------|------------------------------|--------|
| 1 | 0.730 | 25 | |
| 2 | 1.365 | 50 | |
| 3 | 1.450 | 75 | SET I |
| 4 | 1.562 | 100 | |
| 5 | 1.545 | 125 | |
| 6 | 1.669 | 150 | |
| 1 | 0.875 | 25 | |
| 2 | 1.335 | 50 | |
| 3 | 1.450 | 75 | SET II |
| 4 | 1.562 | 100 | |
| 5 | 1.525 | 125 | |
| 6 | 1.630 | 150 | |

The mean concentration of exchangeable Al in the fill samples is:

1.180 mg Al/g of soil sample.

The mean concentration of exchangeable Al in the body samples is:

3.225 mg Al/g of soil sample.

The extractable Al is obtained by extracting the soil with normal ammonium acetate adjusted to pH 4.8 in the same manner described for exchangeable Al. The extractable consists of exchangeable Al, soluble aluminium hydroxide ($\text{Al}(\text{OH})_3$) and probably some hydroxy-Al monomers or polymers which may be strongly adsorbed by the colloid or packed between expansible silicate layers of clay. The same colourimetric measurement as in the case of exchangeable Al was employed to measure the absorbance of the solutions and hence by calculation the concentration of Al present per g of soil sample.

Results: Fill Samples.

| Sample. No. | wt. of sample (g) | Absorbance | mg Al/g of soil |
|-------------|-------------------|------------|-----------------|
| 3651 | 2.5005 | 0.890 | 1.031 |
| 3652 | 2.5065 | 0.995 | 2.064 |
| 3653 | 2.5015 | 0.876 | 1.573 |
| 3658 | 2.4998 | 0.859 | 1.525 |
| 3659 | 2.5019 | 0.995 | 1.538 |
| 3660 | 2.4993 | 0.870 | 1.551 |
| 3673 | 2.5048 | 0.905 | 1.375 |
| 3674 | 2.5033 | 1.080 | 2.356 |
| 3052 | 2.4991 | 0.645 | 1.001 |
| 3053 | 2.5011 | 0.916 | 1.299 |
| 3054 | 2.5026 | 0.760 | 1.018 |
| 3074 | 2.5033 | 0.815 | 1.117 |
| 3075 | 2.3186 | 0.990 | 1.674 |
| 3076 | 2.4964 | 1.045 | 1.545 |
| 3115 | 2.5011 | 1.060 | 1.598 |
| 3116 | 2.4997 | 1.080 | 1.620 |

Results: Body Samples.

| Sample. No. | wt. of sample (g) | Absorbance | mg Al/g of soil | |
|-------------|-------------------|------------|-----------------|-----|
| 3551 | 2.5448 | 1.330 | 2.998 | |
| 3552 | 2.5022 | 1.430 | 3.843 | |
| 3553 | 2.5006 | 1.440 | 3.923 | SET |
| 3554 | 2.5048 | 1.390 | 3.138 | |
| 3555 | 2.5007 | 1.420 | 3.798 | I |
| 3556 | 2.5021 | 1.380 | 3.500 | |
| 3558 | 2.4998 | 1.421 | 3.826 | |
| 3559 | 2.5047 | 1.424 | 3.836 | |
| | | | | |
| 3560 | 2.5004 | 1.295 | 3.474 | |
| 3563 | 2.4920 | 1.442 | 4.529 | |
| 3564 | 2.5002 | 1.330 | 3.725 | SET |
| 3589 | 2.4997 | 1.240 | 3.001 | |
| 3590 | 2.5011 | 1.265 | 3.247 | II |
| 3593 | 2.5035 | 1.405 | 4.238 | |
| 3595 | 2.5007 | 1.600 | 4.523 | |
| 3600 | 2.5001 | 1.305 | 3.550 | |

Volume of 25ml leachate used is 10ml.

Mean Al concentration for body samples is: 3.696mg Al/g of soil.

Mean Al concentration for grave fill samples is: 1.493mg Al/g of soil.

Calibration data using standards:

Body:

| Volume of 25ppm Al std. used/ml | Absorbance | mg Al/50 ml soln. | |
|------------------------------------|------------|-------------------|--------|
| 3 | 1.130 | 75 | |
| 6 | 1.285 | 150 | SET I |
| 9 | 1.520 | 225 | |
| 12 | 1.756 | 300 | |
| 2 | 0.575 | 50 | |
| 4 | 1.095 | 100 | |
| 6 | 1.255 | 150 | SET II |
| 8 | 1.395 | 200 | |
| 10 | 1.530 | 250 | |
| 12 | 1.660 | 300 | |

Grave fill:

| Volume of 25ppm Al std. used/ml | Absorbance | mg Al/50 ml soln. |
|------------------------------------|------------|-------------------|
| 1 | 0.645 | 25 |
| 2 | 0.760 | 50 |
| 3 | 0.910 | 75 |
| 4 | 0.970 | 100 |
| 5 | 1.125 | 125 |

The concentration of Al was expressed as mg Al/g of soil sample and in the case of the extractable Al the concentration in both the grave fill and body region samples were found to be fairly constant. The average concentration of extractable Al in the body was 3.696 mg Al/g of soil, and that in the grave fill was 1.493 mg Al/g of soil. The average of the exchangeable Al was 3.225 mg Al/g soil in the body, and 1.180 mg Al/g soil in the fill. Due to this large difference in concentration it was decided to try and find a test to show up the presence or absence of a body in the soil by use of its Al content. The test employed was engineered as follows:

- a) 3ml ammonium acetate (pH 4.64) was added to a spatula full of soil in a test tube.
- b) The mixture was shaken for about 1 minute and allowed to settle.
- c) 5 drops of the supernatant were transferred to a clean test tube.
- d) 2 drops of Eriochrome Cyanine dye were added to the supernatant followed by 2 drops of ammonium acetate buffer (pH 6.0) and the purple-red colour allowed to develop.

The test was repeated using KCl instead of ammonium acetate as the leaching agent.

| Sample No. | Leaching time (m) | Time taken for colour to develop (s). | Leaching agent. |
|------------|-------------------|---------------------------------------|---------------------|
| 3589 | 10 | 1 | NH ₄ OAc |
| 3674 | 10 | 10 | NH ₄ OAc |
| 3589 | 1 | 10 | NH ₄ OAc |
| 3674 | 1 | 60 | NH ₄ OAc |
| 3589 | 10 | 10 | KCl |
| 3674 | 10 | 20 | KCl |
| 3589 | 1 | 20 | KCl |
| 3674 | 1 | -- | KCl |

3589 = body sample

3674 = fill sample

From these results it was concluded that using KCl as a leaching agent removes very little exchangeable aluminium from the soil. It removes enough from the body region (where there is a high concentration of Al) to be detected by the above procedure, but not enough from the grave fill region (where there is a low Al concentration). This method is definitely not the most sophisticated of methods but at present it is the only one which will not alter the soil in any manner. In the case of a spray it would probably interfere with other constituents of the soil besides the Al and alter the composition of the soil. More work will be done on this aspect in the future.

Ref.

Black, C.A., Evans, D.D., White, J.C. and Ensminge, L.E. (1965)

Methods of Soil Analysis, pt. 2: Chemical and Microbiological Properties pp. 985-991.

Sutton Hoo - Leverhulme Trust Project

Comparative Material - Atlantic Trading Estate, Barri, South Glamorgan.

ATE 87.

ATE is an Early Christian monastic and settlement site in South Wales, with an equivalent date range to the post-Roman/early Saxon period in England. The site is being excavated by the Glamorgan-Gwent Archaeological Trust, whose kind permission was given to sample one of the graves. Samples taken from this site were collected on 17th-19th March 1987, by PHB. Thanks are due to Richard Newman and his staff.

The samples were from ATE site 92, Inhumation no. 28, Feature no. 11, Context nos. 441 (skeleton) and 449 (grave fill).

The inhumation was an extended flat burial, oriented SW-NE, with the head to the SW. The fill consisted of a light coloured medium sand, of a very consistent, homogeneous nature, containing almost no gravel/stones. It appeared very much like a beach sand, which is in fact what it originally was. The inhumation was one of an irregular row running roughly N-S along the junction of the beach sand and the overlying 'burgundy'-coloured silt-sand. In fact this overlying layer had been eroded to reveal some of the inhumations. The site of the burials is now some distance from the sea, but presumably lies approximately at the early medieval shoreline, since extended by land filling. Like most of the other graves, erosion was worst on the SW side, and indeed there was very little fill remaining at the head end. However, the grave did not appear to have been disturbed, and there was a reasonable depth of grave fill remaining at the NE end.

The skeleton uncovered and sampled was almost complete, with clear ribs, and finger bones surviving (although the hands were crossed over the pelvis). No toe/foot bones were recovered. However, despite the superficially good preservation, the bones looked 'weathered' and pitted. Most of the epiphyses had disintegrated, and the surviving bone was very fragile and brittle. The excavators reported that this particular skeleton was in a generally worse state of preservation than the norm. There is consistently enough skeletal evidence for ageing, sexing, and pathological examination. Compared to Sutton Hoo, the preservation is remarkably good. Many of the burial conditions appear at first glance to equate with those at Sutton Hoo, but at ATE there has been no cultivation, and hence no addition of chemical fertilisers; the overlying 'burgundy' deposit (so-named by the excavators) may have extended over the burials, and hence served to seal them and retard dissolution; the pH of the sand is not yet known, but the presence of visible fragments of shells, etc, suggests that the soil veers towards the alkaline.

Note on sampling procedure:

Following the suggestions put forward in the project interim report, It was decided to use a different pattern of sampling than that employed at SH 86. A series of sample points 10 cm apart was marked along a line running along the main axis of the burial. It must be pointed out that this was

quite hard to locate properly at the beginning - the grave cut was impossible to distinguish visually. This resulted in the main axis line being slightly out of alignment with the actual body axis. However, this did not prove to be too much of a disadvantage. Originally, two lines of samples at right angles to the main line were taken. A further line of three was added in order to recover more material around the body. This arrangement is shown in the diagramme below. (The samples were 10 cm apart horizontally, and 5 cm apart vertically). The sampling pattern was chosen particularly for speed - in practice, alternate 'boxes' were removed, down to the body level, and the samples taken from the resulting section faces. This made things much quicker, and made measurement of the samples much more accurate, as there was no necessity to keep levelling every sample. Once the body level had been reached, and the samples taken, the upstanding 'blocks' were removed to expose the skeleton for recording. On removal of the skeleton, the sample position pattern was reestablished, and a further set of samples taken below the burial level, using the same technique of 'box' removal and sampling from the sections. There was a small amount of confusion over the levels of some of this lower series, which resulted in the duplication of some samples. The extra line of samples running N. of the main axis (points 21, 22, and 23) were added to give more samples from the body area, once it was realised that the main axis was slightly misaligned. This misalignment was in fact not a bad thing, as it produced a number of samples from well beyond the grave (those running away to the S), and so 'background' material was included in the original sample. It was partly because of this, and partly due to time, that a background column was not taken at ATE. It was felt by the sampler that such material could be collected later if necessary, as the scale of 'body' material movement under the prevailing conditions could be assessed from the material recovered. The basic data base thus consists of 23 columns of samples, of varying depth, from the extant top of the grave fill to below the burial.

Comparative Material: Brandon, Suffolk.

Following our first visit last year, a subsequent visit was made on June 10th 1987. There were no inhumations exposed at this time, but sampling was carried out on two post holes. Both were revealed in half-section, and sampling was carried out from the exposed section faces. The two features sampled were contexts 7980 and 7981.

7980: Was a post hole, with the remains of the wooden post clearly visible in the centre, as a narrow vertical band, tapering at the bottom, of very dark brown/black silty sand, with a high percentage of fragments of charcoal-like organic matter. The dimensions of this were approx. 0.06m wide x 0.35m deep. Surrounding the dark central wood 'stain', was a narrow pale grey-brown 'halo', approx. 0.01-0.02m wide. The post remains were set in a bright yellowish-brown sandy backfill.

7981: Was a slightly different type of feature, basically being a vertical section through a plank 'stain', running E-W: ie. the visible section was perpendicular to the long axis of the plank. The 'plank stain' appeared as a rectangular light brown patch in the yellowish-brown backfill. It measured approx. 0.40m vertically x 0.08m horizontally.

Sampling was kept as simple as possible, to give a line of samples from some distance outside the organic residues, into the centre of the residue, and if feasible, out the other side again! This is best seen on the accompanying diagram. A total of 48 samples were taken from both features, and were put into clean Minigrip bags on site. They were then dried and bottled as soon as possible.

Comparative material: Snape, Suffolk.

Suffolk County SMR code SNP 007.

As the notes/interim provided by the excavator, William Filmer-Sankey, relate, Snape is of great importance as a comparison to Sutton Hoo, because of the discovery of another ship burial. Conditions at the site are also very comparable to Sutton Hoo, as it is only a few miles away.

Still within the Sandlings province, the archaeology lies very close to the topsoil-stripped surface. In fact, it is nearer the surface than at SH, as there has been plough damage to some of the cremations. A very similar depth of plough soil is visible at Snape, ie. 0.30-0.35m. The subsoil has quite a different appearance to that at SH, however, being much more disturbed by root action (presumably bracken). The soil is very sandy, and appears very homogeneous. It is of a noticeably ash-grey colour, compared to the brighter yellow of SH. Very numerous 1cm. dark brown root holes can be seen.

WF-S reports that the inhumations had degraded to silhouettes, without any trace of bone recoverable. This seems to be even more of an extreme of preservation than at Sutton Hoo, where only one burial was found to be totally devoid of bone fragments.

The finds were sampled by the excavators, and appear to be larger than the average sample taken by PHB, and not so precisely located. However, they are valuable in providing a body of suitable silhouette material for comparative study.

Note on sampling procedure for trace-element analysis.

Following discussion of the sampling procedure after the first field season at Sutton Hoo, the grave sampling technique was altered. It was basically the same, but rather more controlled. The sample array was set out as depicted below (Fig. 3), with a line of points running along the main axis of the grave at 10 cm intervals, with two perpendicular lines positioned to divide the grave approximately into thirds. It is important that the lines of points run beyond the limits of the grave, so that comparative samples from the surrounding natural are taken as 'control'. Vertical sample interval was 5 cm.

It was discovered that excavation was much quicker using the 'box' system, especially at Sutton Hoo, where the recording system requires the drawing of the cumulative section, and the planning of each 10 cm 'spit'. With the sample lines dividing the grave into a series of 'boxes', (Fig. 3), the procedure was as follows:

- 1) B, D and F are excavated to a depth of 10 cm. (This enables both longitudinal and transverse sections to be drawn, if desired).
- 2) Samples are taken at the set points along the various lines, both at the surface, and 5 cm. below the surface, from the face of the section.
- 3) A, C, and E are excavated to a depth of 10 cm, leaving a horizontal plane 10 cm below the surface of the grave. (This can then be planned/photographed).

These three basic steps are repeated through the bottom of the grave, to a depth of 10 cm below the level of the body.

Of course, this is the idealised version of the procedure. In practise, there are several other points which need consideration.

- a) The grave surface is naturally not always horizontal. The use of a datum line is thus recommended, and true horizontal levels established as one proceeds down the grave. This may mean that one end of the grave has taller sample columns, but it is very important that samples are taken from the same horizontal level throughout the grave, if they are to be compared.
- b) The sides of the grave cut may not be vertical. This of course means that some sample columns may hit natural after only one or two vertical intervals, if the sides of the cut slope inwards. This does not matter, and cannot be altered at the expense of the removal of fill material in its proper stratigraphic sequence. Of course, those points on the surface lying outside the grave cut will only be sampled at the surface - the 'box' excavation does not extend beyond the feature limit.
- c) The body always gets in the way. It can be a problem to reconcile the exposure of the body/skeleton with maintaining the integrity of the sample array. However, in practise, it is not so difficult. At Sutton Hoo, where any sample points falling within the 'sandmen' were treated

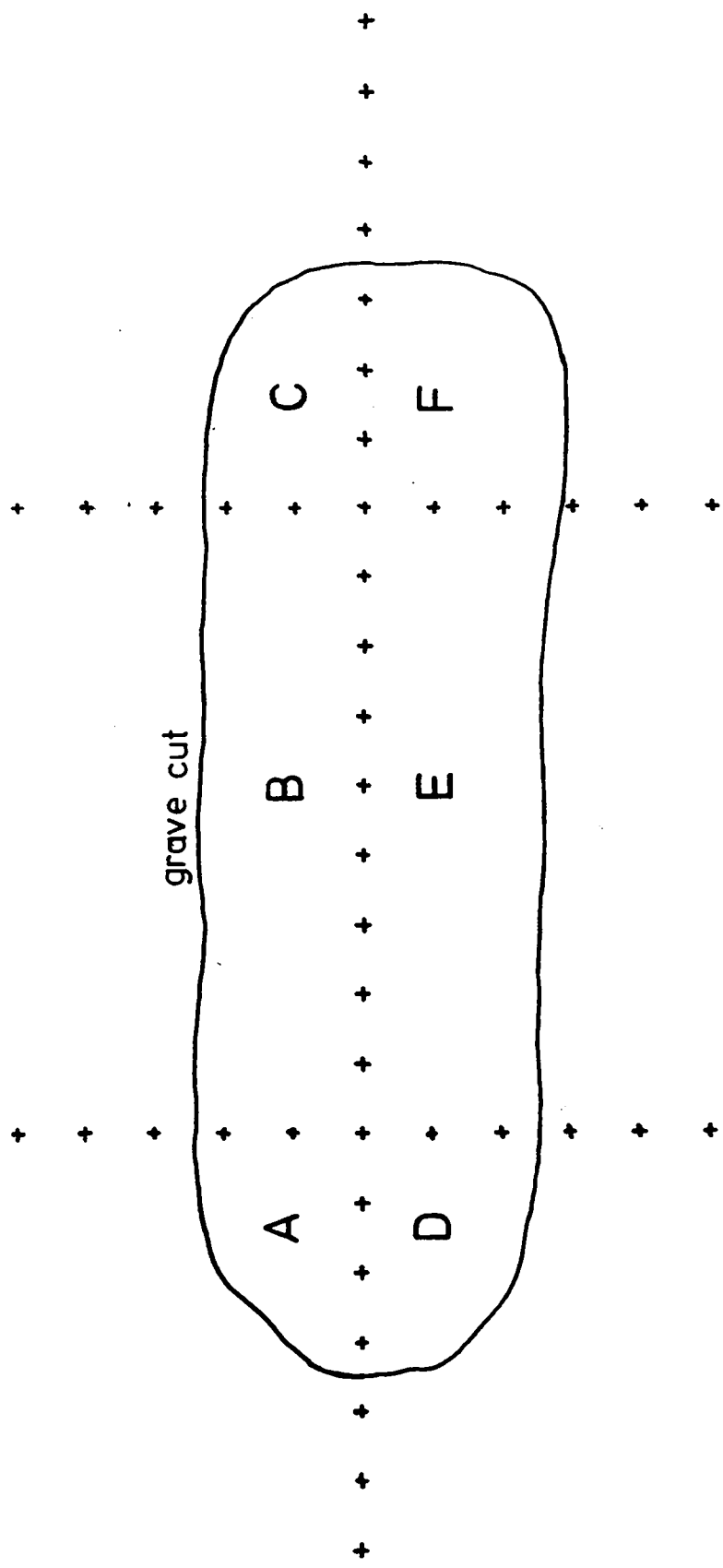


Fig. 3- Revised sampling array, showing "boxes".

as normal, the amount of material removed did not alter the appearance of the final tableau. Where a skeleton is being excavated, (as at Atlantic Trading Estate), material can usually be removed from around and adjacent to the bones, without disturbing them. As most burials occupy a very limited vertical space, ie. only one or two vertical intervals, and as only a certain proportion of the sample points actually coincide with the body, the problem is not a serious one. When the body has been exposed for drawing/photography, any samples can be taken at the same time as the body is removed.

The actual samples consist of c. 30 g of soil, collected with a stainless steel spatula, into a clean pre-numbered minigrip polythene bag. Naturally careful labelling must be practised. The hardest part of the procedure at Sutton Hoo was the repositioning of sample points after removal of tapes and strings etc. for photography of the various spit levels. The use of a datum line with fixed end points, graduated strings (with marks at 10 cm intervals) and a plumb bob, all these problems were overcome. Once the grid reference of the top points were established, all subsequent points below them had the same grid reference - there was no need to re-measure the XY-coordinates. So a simple numbering of the initial points, followed by a number for each 5 cm level, gave a quick method of uniquely labelling each sample in the field, easily converted to the real XYZ coords. after excavation. A further point on accuracy of location of the samples, is that by dint of the samples being more than a grain of sand each, the actual location is no more than the central locus of each sample. Hence some leeway is permitted, for example, if there is a stone at the precise sample point, soil can be collected from around it. The only really important point to bear in mind, is that samples must be separated from all the others around. In practise, there was a tendency for samples not to be taken from a uniform distance around the location point - as the horizontal distance between points was greater than the vertical, so the horizontal 'catchment area' of each sample tended to be greater than the vertical.

This basic sampling procedure is valid for any soil type, and is to be recommended when sampling any feature for the purposes of trace-element or other analysis. Where recording of 10 cm spits is not required, the depth of excavation at stage (1) of the excavation procedure above can be increased as far as the soil type will support the sections, or the samples can be conveniently taken, thus enabling rapid sampling.

Sutton Hoo - Leverhulme Trust Project

Summary of expenditure, from Mar. 87 to end Feb. 88.

110 - Equipment and materials.

| | | Total |
|-------------------------|-----------|---------|
| 10 - Plastic bags | - £ 12.42 | £ 12.42 |
| 11 - Photographic costs | - £ 9.36 | £ 21.78 |

300 - Office and Admin.

| | | Total |
|-------------------------------------------------|------------|----------|
| 05 - Contribution to central office costs 87/88 | - £ 300.00 | £ 300.00 |

084 - Student's chemicals/consumables.

| | | Total |
|------------------------|-----------|----------|
| 02 - Chemicals | - £ 61.44 | £ 61.44 |
| 03 - Chemicals | - £ 8.51 | £ 69.95 |
| 04 - Chemicals | - £ 6.56 | £ 76.51 |
| 05 - Chemicals | - £ 5.06 | £ 81.57 |
| 06 - Pipette microtips | - £ 18.98 | £ 100.55 |
| 07 - Chemicals | - £ 12.42 | £ 112.97 |
| 08 - Ribbons/discs | - £ 11.43 | £ 124.40 |
| 09 - Syringe | - £ 28.75 | £ 153.15 |
| 10 - Cylinder rental | - £ 12.54 | £ 165.69 |
| 11 - Cylinders (gas) | - £ 57.50 | £ 223.19 |
| 12 - Amberlite resin | - £ 37.95 | £ 261.14 |
| Total | - £ | £ 261.14 |

320 - Travel and conferences.

| | | Total |
|------------------------------------------------------|------------|-----------|
| 23 - Conference fee, Edinburgh. | - £ 18.00 | £ 18.00 |
| 24 - Subsistence, travel, etc. to Edinburgh. | - £ 60.00 | £ 78.00 |
| 25 - Travel, subsistence, to Barry, site sampling. | - £ 77.68 | £ 115.68 |
| 26 - Fees, travel, subsistence SH seminar in Oxford. | - £ 63.80 | £ 219.48 |
| 27 - J. Miles to Oxford C14 lab., analysis. | - £ 50.00 | £ 269.48 |
| 28 - Expenses to central travel (Unit car) 87/88 | - £ 400.00 | £ 669.48 |
| 29 - Return journey from Birmingham to Sutton Hoo | - £ 22.90 | £ 692.38 |
| 30 - Interview expenses, L. Stewart | - £ 22.70 | £ 715.08 |
| 31 - Travel costs for visit to Brandon & Egham | - £ 41.57 | £ 756.65 |
| 32 - Vehicle hire - van to Sutton Hoo, petrol, etc. | - £ 211.34 | £ 967.99 |
| 33 - Conference fee and accomodation, Glasgow | - £ 61.60 | £ 1029.59 |
| 34 - Petrol | - £ 36.35 | £ 1065.94 |
| 35 - Travel costs return Birmingham to SH | - £ 26.00 | £ 1091.94 |
| 36 - Rail travel to Glasgow conference | - £ 33.00 | £ 1124.94 |

| | | |
|------------------------------------------|-------------|-----------|
| 37 - Petrol | - £ 13,80 | £ 1138,74 |
| 38 - Vehicle hire - transport of samples | - £ 128,98 | £ 1267,72 |
| Total | - £ 1267,72 | |

416 - Research Associate Salary

| | | Total |
|---------------------------|-------------|-----------|
| 19 - Gross salary Mar, 87 | - £ 1628,86 | £ 1628,86 |
| 20 - Gross salary Apr, 87 | - £ 1151,87 | £ 2780,73 |
| 21 - Gross salary May 87 | - £ 1151,87 | £ 3932,60 |
| 22 - Gross salary Jun, 87 | - £ 1151,87 | £ 5084,47 |
| 23 - Gross salary Jul, 87 | - £ 1151,87 | £ 6236,34 |
| 24 - Gross salary Aug, 87 | - £ 1151,87 | £ 7388,21 |
| 25 - Gross salary Sep, 87 | - £ 1151,87 | £ 8540,08 |
| 26 - Gross salary Oct, 87 | - £ 1151,87 | £ 9691,95 |
| 27 - Gross salary Nov, 87 | - £ 1151,87 | £10843,82 |
| 28 - Gross salary Dec, 87 | - £ 1151,87 | £11995,69 |
| 29 - Gross salary Jan, 88 | - £ 1157,87 | £13147,56 |
| 30 - Gross salary Feb, 88 | - £ 1157,87 | £14299,43 |
| Total | - £14299,43 | |

413 - Student fees/payroll

| | | Total |
|---------------------------------------|-------------|-----------|
| 07 - Grant for JUM Jan, 87 | - £ 229,67 | £ 229,67 |
| 08 - Grant for JUM Feb, 87 | - £ 229,67 | £ 459,34 |
| 09 - Grant for JUM Mar, 87 | - £ 229,67 | £ 689,01 |
| 10 - Grant for JUM Apr, 87 | - £ 229,67 | £ 918,68 |
| 11 - Grant for JUM May 87 | - £ 229,67 | £ 1148,35 |
| 12 - Grant for JUM Jun, 87 | - £ 229,67 | £ 1378,02 |
| 13 - Grant for JUM/LJS Jul, 87 | - £ 459,34 | £ 1837,36 |
| 14 - Fees for LJS as temp. student | - £ 1165,00 | £ 3002,36 |
| 15 - Grant for LJS Aug, 87 | - £ 229,67 | £ 3232,03 |
| 16 - Grant for LJS Sep, 87 | - £ 238,25 | £ 3470,28 |
| 17 - Grant for LJS Oct, 87 | - £ 238,25 | £ 3708,53 |
| 18 - Grant for LJS Nov, 87 | - £ 238,25 | £ 3946,78 |
| 19 - Grant for LJS Dec, 87 | - £ 238,25 | £ 4185,03 |
| 20 - Fees for LJS as o/seas student | - £ 4935,00 | £ 9120,03 |
| 21 - Grant for LJS Jan, 88 | - £ 238,25 | £ 9358,28 |
| 22 - Grant for LJS Feb, 88 | - £ 238,25 | £ 9596,53 |
| 23 - Fees for PHB M.Phil registration | - £ 180,00 | £ 9776,53 |
| Total | | £ 9776,53 |

Grand totals:

| | |
|-------|-----------|
| 084 - | £ 261.14 |
| 110 - | £ 21.78 |
| 300 - | £ 300.00 |
| 320 - | £ 1267.72 |
| 413 - | £ 9776.53 |
| 416 - | £14299.43 |
| Total | £25926.60 |

Total 85/Feb. 87 £23292.88.

Total expenditure £49219.48.

Grant remaining £26678.52.