# Soil phosphorus levels at archaeological sites

*by J C McCawley and H McKerrell*

Phosphate tests and the possibility they offer for detecting ancient settlements and the presence of man in cultural layers have been used often and reported in detail (for summaries

see Cook and Heizer 1965, 1; Provan 1971, 37). The use for detailed work such as recognising skeletal remains has found perhaps less use but has often proved successful (Solecki 1951; Johnson 1956).

Over the past few years the National Museum's Research Laboratory has determined the phosphorus content of a number of soils from various Scottish archaeological sites (mainly sepulchral) where little or no evidence was visible of bodily remains or human activity. Essentially the method is to contrast the phosphorus content of archaeologically significant soils with background control samples. Where these latter provide typical low phosphorus levels and the former much higher levels, we can usually be confident that the high levels are due to some relevant prehistoric activity. In general, when the results do not conform to this pattern no useful conclusions can be drawn.

Phosphorus occurs naturally in soils in both inorganic and organic forms, the amounts and proportions of each varying widely. At the soil surface phosphorus-containing minerals are subject to weathering and to attack by micro-organisms, which results in the release of phosphorus mainly as orthophosphates. These phosphate ions can be leached downwards by surface water, or taken up by plants. The latter does not result in an irretrievable loss as the dead plant gives back the phosphorus, now organic, to the natural phosphorus reservoir. The reservoir remains fairly constant, with higher concentrations at the surface, unless added to by human activity. These additions can be summarised as: (1) from skeletal remains in burials; (2) from the urine and faeces of man and animals around habitation areas; (3) from food refuse; (4) from animal manure and phosphorus based chemicals used as fertilisers. It is with the first that we are particularly concerned.

Bone is a polycrystalline material composed of an inorganic and organic fraction. The inorganic fraction, accounting for approximately three quarters of bone, is composed chiefly of the phosphorus containing mineral hydroxyapatite. The organic material of bone and of body tissue, flesh, etc, will contain a little phosphorus but the main source of phosphates in a burial will be the skeleton (630 g of phosphorus is contained in the body of an 11 stone man, 80% of which is in the skeleton) (Fearn 1948, 143).

The bulk of the evidence points to phosphates being retained in the majority of soils. Various mechanisms have been proposed and it is probable that there is a combination of chemical-bonding, ion-exchange, and adsorption processes participating in the fixation of phosphorus. Strongly bonded, insoluble compounds having restricted mobilities are quickly formed with many soil constituents. Lysimeter experiments have shown that phosphates are leached only slowly from the majority of soils. Heizer summarises as follows: in soils on the alkaline side of neutrality (pH 7 to 14) phosphorus is leached relatively slowly. When the pH is below 5 (pH 1 to 5) the leaching effect is also slow. At pH conditions of only slight acidity (pH 6 to 7) the phosphates are leached relatively quickly (Heizer and Cook 1965, 13). The amount of phosphorus retained will depend upon soil pH, temperature, moisture availability, the concentration of such minerals as iron, aluminium and calcium, and the time elapsed since burial.

*Results*

A selection of results from various sites are presented below. Typical results are illustrated in fig 5.

| *Site* | | *pH* | *Phosphorus ppm** | |
|---|---|---|---|---|
| | | | *Background* | *Site samples* |
| Limefield, Lanarkshire, EBA cairn | | 6·2 | 200, 250 | 600, 1500, 900 |
| Strathairlie 2, Fife, EBA cist | | | 100 | 100, 200, 400, 350, 150 |
| Harelaw, Lanarkshire, 1968, BA cairn | | 6·2 | 100, 450 | 200, 450 |
| Harelaw, Lanarkshire, 1969, BA cairn | | 6·4 | 100, 200 | 1000, 2000 |
| Cairneyhill, Lanarkshire, BA cairn | | | 2000 | 4000, 4000, 4000 |
| Whitekirk, E Lothian, IA habitation | | 6·2 | 2000, 4000 | 2000, 4000, 4000, 4000, 3000 |
| Lamington, Lanarkshire, BA ring ditch and enclosure | | | 1000 | 1000, 700 |
| Kinneil Mill, Stirlingshire, BA ring ditch with urns | | 6·8 | 200 | 400, 1500 |
| Dunfermline, Aberdour Road, Fife, BA cists | cist 2 | 6·2 | 200, 300, 300 | 700, 900, 900 |
| | cist 3 | 6·2 | 100 | 600, 1000, 3000 |
| Corney Fell, Cumberland, cairn | | 6·4 | 300 | 500, 1200, 1200, 1000, 600 |
| Barnes Farm, Dalgety Bay, Fife, short cists and wooden coffin graves | grave (5) | 7·1 | 200 | 2000, 1300, 2400, 2400, 3000 |
| Almond Bank, Perth, BA cists | cist V | 6·4 | 2000 | 2000 |
| | cist X | 6·4 | 1800 | 1800 |

*ppm – parts of phosphorus per million parts of air dried soil.

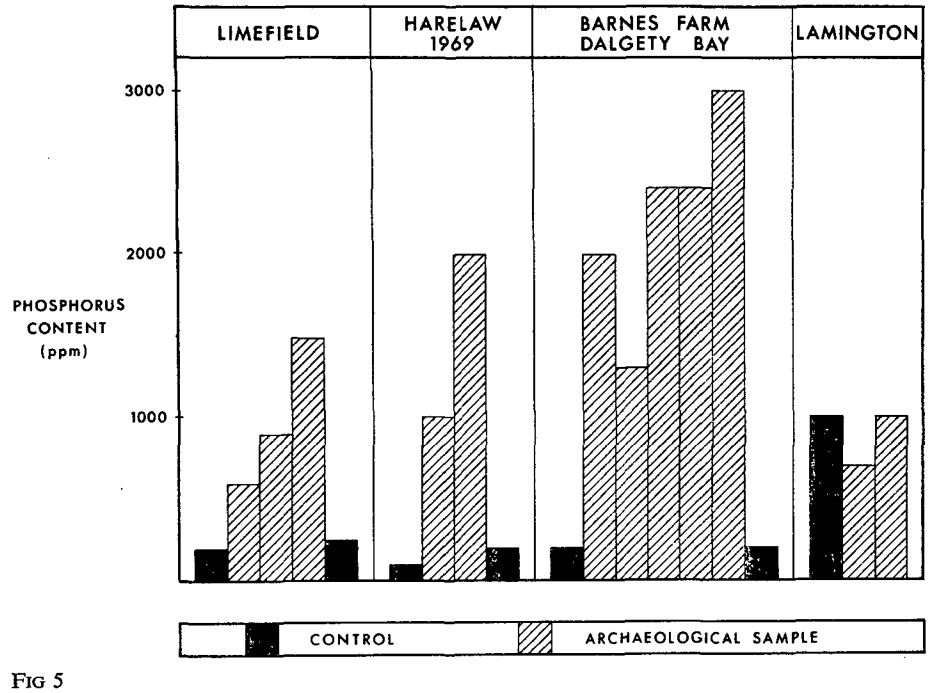


FIG 5

Normal background levels are typically 100–200 ppm of phosphorus and on this basis the excavated soils from Limefield; Harelaw 1969; Kinniel Mill (Marriott 1968); Aberdour Road, Dunfermline; Corney Fell, Cumberland; and Barnes Farm, Dalgety Bay, are indicative of bodily remains. A similar low background level was present at Strathairlie, where teeth enamels were discovered at the excavation site. Samples here were taken diagonally across the cist and the results clearly show a peak in the phosphorus levels at the cist centre, again indicative of bodily remains. Background levels from Cairneyhill, Whitekirk, Lamington, Harelaw 1968, and Almond Bank, Perthshire, are all higher than normal and it is perhaps no coincidence that all of these

sites provided archaeological soils with little significant increases in phosphorus content above background – either extensive prehistoric or, as is more likely, modern contamination (cattle, sheep, fertilisers, etc) has blurred the differences that might have been expected.

The soil pH where measured has, with the exception of the site at Barnes Farm, fallen between 6 and 7; this is the range in which phosphorus is leached most rapidly and no skeletal remains were found at these sites. Barnes Farm, Dalgety Bay has a pH (7·1) on the alkaline side of neutrality and skeletal material at this site had survived. The grave examined here had a distinct body-shaped stain containing bone fragments.

*Analysis*

A sample of the soil was air dried and roughly sieved to remove stones, root material, etc. A small sample, typically half a gram, is taken for the analysis and a standard colorimetric technique to measure total phosphorus, based on the formation of a yellow phosphomolybdovanadic acid, is applied (Stanton 1966, 77). Results are expressed as parts of phosphorus per million parts of air dried soil.

*On-site analysis*

Often the time which elapses between sampling and laboratory analysis can be critical to the archaeologist and emphasises the occasional need for on-site analysis. Such a test developed by Grundlach (1961) and simplified by Schwarz (1967) has been modified slightly and used successfully by this laboratory. The method as used by Schwarz is based upon the formation of a blue coloured phosphorus complex by the addition of two reagents to a small sample of the soil placed on a filter paper. To simplify the method a little more for field use, the following procedure was adopted.

Filter paper (diameter 9 cm) was soaked in solution A, allowed to dry, and stored in polythene bags. When used for testing for phosphorus a small sample (0·1 to 0·5 g) was placed in the centre of the filter paper and three drops of solution B added. When phosphorus is present a blue stain is formed which spreads outwards from the centre of the paper. The amount of phosphorus present is shown by the extent and intensity of the stain. Calibration can be achieved by using soils of known phosphorus contents and recording the intensity and extent of the molybdenum blue formed. As the technique is purely for comparative use, no such calibration was carried out in this laboratory. The procedure was used by Dr T F Watkins at the excavation at Barnes Farm, Dalgety Bay. Samples taken at regular vertical distances during the uncovering of a wooden coffin grave indicated strikingly, by high phosphorus levels, the body level and allowed a greater degree of care to be taken.

*Solution A*

5 grams of ammonium molybdate is dissolved in 100 millilitres of cold water; after solution 35 millilitres of concentrated nitric acid (specific gravity 1·4) is added.

*Solution B*

0·5 solution of ascorbic acid in water.

The impregnated filter papers were found to be usable up to five days after preparation. After this time breakdown of the impregnating materials certainly occurs and produces spurious blue colourations. When possible papers should be prepared for use daily to obtain the best possible results.

Plate 43 shows the kind of results obtained and the equipment used.

*Sampling*

The number and separation of samples is obviously dependent upon the extent of the area of interest and the kind of information required. Chemical surveying of, for example, a suspected settlement will not require closely spaced samples but perhaps one every 15 to 30 feet. A smaller area such as a cist or grave presents little problems as to where to sample. In a grave suspected of once having contained a body samples are sensibly taken at short distances (every 2 ft) down the longest axis (length) and similarly across the breadth through the centre. This covers the likely area of occupation and gives a profile in two directions. Of great importance is the taking of control samples. At a burial site these should be taken from just outside the grave area and also from a further distance away (15 to 30 ft), depending upon the layout of the site. Where possible the control sample should be taken at the same level as the site samples as natural phosphorus levels differ with the distance from the surface. If a number of samples are being taken their locations are best recorded on a site diagram using a vertical and horizontal axis drawn from some imaginary point outside the main area. Similarly samples from different levels can be recorded in the same way.

Sample size is not critical but is ideally 200 to 300 grams (a large handful) to allow the possibility of further analysis and to give an idea of colour, texture, etc. Storage is best made in self seal polythene bags which can themselves be written on. Information usefully included is sample location, site reference, colour or texture changes during excavation, and the nature of the enquiry.

*Manganese*

A silhouette of a skeleton in a 'coffin' was revealed during excavations at Bishop's Waltham, in Hampshire. Analysis showed no accumulation of phosphate in the 'body' compared to the 'neutral' earth around. However, manganese had accumulated in the chocolate-brown body, and the coffin, to levels a hundred times greater than those outside the grave area (Ashbee 1957). This accumulation of manganese in areas of high organic content has been investigated in this laboratory. Samples suspected of being a 'body', wood, leather, etc have been examined and, although insufficient material has as yet been looked at, certain promising trends have appeared. It is the intention that other metals, such as copper and aluminium, should also be surveyed in this way.

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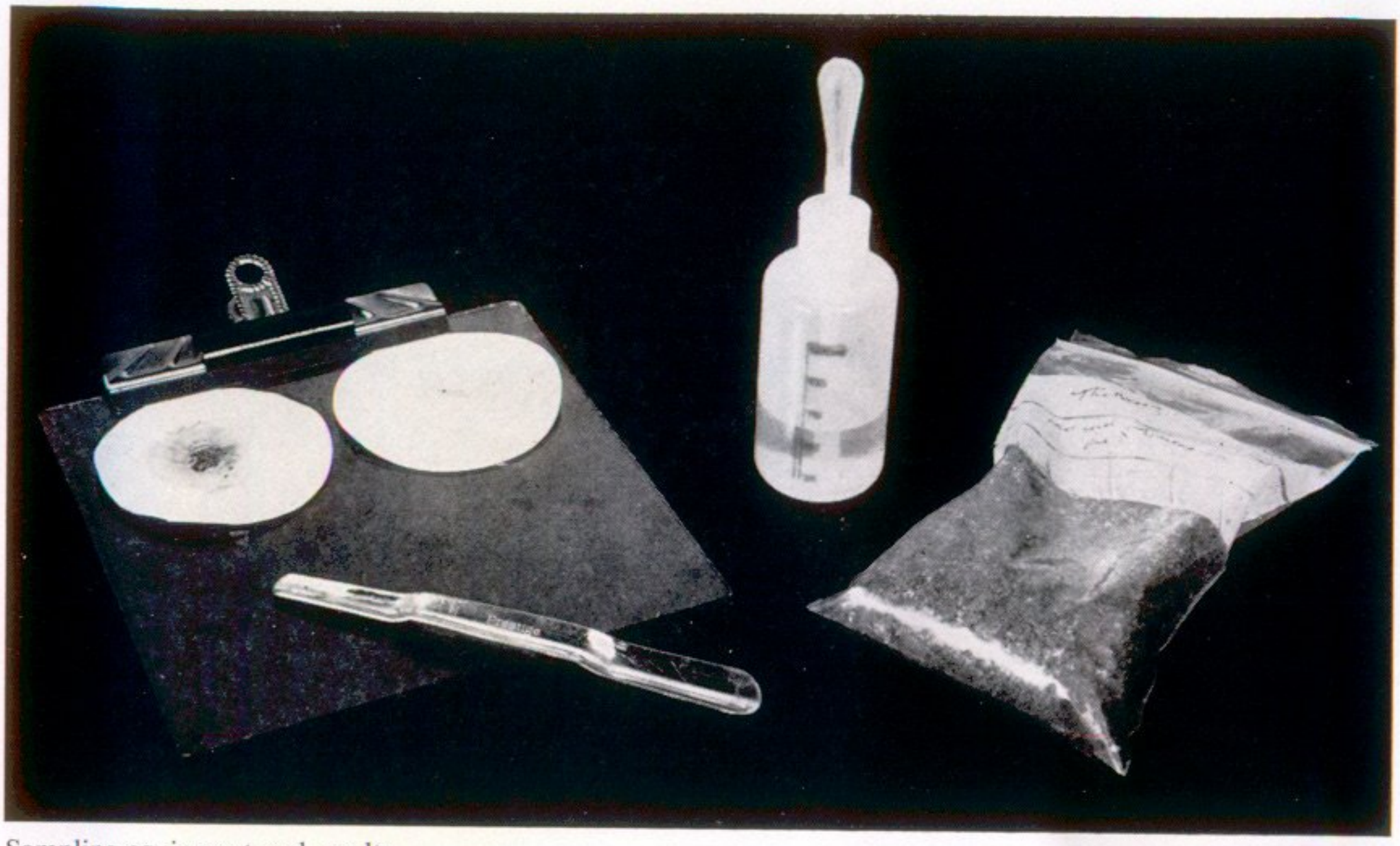

Sampling equipment and results