Report for 1975 - Part 1

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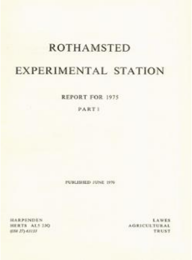

ROTHAMSTED

EXPERIMENTAL STATION

REPORT FOR 1975

PART 1

Pedology Department

C. Bloomfield

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Introduction

Our mineralogical work included the continuation of the study of interstratified clay minerals, and of the mineralogy of loess soils. We are also studying synthetic illite-like compounds, in collaboration with Professor B. Velde of The University of Paris. Work on the weathering of mica in soils has revealed that a mixed-layer mica-smectite is the main product, and not vermiculite, as was previously thought.

We have examined soils from the Solomon Islands on behalf of the Land Resources Division of the Ministry of Overseas Development. One of these soils contains sufficient bauxite for it to be a potential economic source.

Collaboration with The International Institute of Tropical Agriculture in the study of the mineralogy of Nigerian soils continued; bastnaesite, a rare earth fluorocarbonate, was found in the weathered parent rock of one of these soils.

Infra-red spectrometry has been shown to provide a method for determining quartz and kaolinite that has advantages over selective dissolution and X-ray diffraction methods.

As part of a collaborative study of soil structure, we are measuring the changes in the pore size distribution that occur when clay soils are air-dried.

In our geochemical work we have continued to study the distribution of minor elements between soil constituents; work on the geochemistry of the soils of Pembrokeshire, which is now finished, included a study of the distribution of Br and Pb in the soils and herbage of the area.

Collaboration with IITA in studying the effects of cropping and management on the organic matter and nutrient reserves of tropical soils continues, as does our investigation of the effects of air-drying on metabolism in soil. Determination of the organic matter contents of soils taken from the Hoosfield Permanent Barley experiment during the period 1882–1975 showed that equilibrium between the gain and loss of organic matter by the farmyard manure-treated soil has not yet been attained.

Work continued on the non-colloidal metal-complexing products of the decomposition of plant matter in soil, and on the chemical properties of heavy metals in contaminated sewage sludge. This aspect of our work was recently extended to deal with the fixation of Cu in peat soils.

Mineralogy

Interstratified minerals. We have continued our study of the interpretation of X-ray diffraction patterns of interstratified clay minerals. The experimental X-ray diffraction pattern can often be fitted quite closely if parameters are arbitrarily adjusted in a model structure of two or three layer-types based on known clay minerals, with variable proportions of layers, and variable chances that one type of layer will succeed another. In some cases we have combined this approach with the direct calculation, by Fourier transform from measured intensities, of the chance that layers are to be found at particular distances apart. This treatment is only exact if the layers all have the same distribution of electron density when projected on to a line perpendicular to the clay mineral layers. This is approximately true if both components of the interstratification are layer silicates of the same type, say 2:1, and the interlayer material has only a small fraction of the total electron density.

An air-dry sodium-saturated clay from the Old Red Sandstone from Monmouthshire gave an X-ray diffraction pattern that showed an interstratified phase with a first spacing of 11·1 Å, and a small quantity of chlorite. Ignoring reflections due to the chlorite component, the Fourier transform method gave a distribution curve for layer-to-layer distances with prominent maxima at 10, 12·5, 20 and 22·5 Å that could be interpreted as a mixture of 39% 12·5 Å and 61% of 10 Å layers stacked so that there was a chance of 0·23 that a 12·5 Å layer would be followed by another of the same spacing. The distribution curve also showed smaller peaks and some pairs of positive and negative peaks that are probably mainly due to the error involved in the assumption that all the layers have the same structure. To check whether these errors affect the results, we calculated the diffraction pattern that would be obtained for a model structure with 12·5 Å sodium smectite and 10 Å illite components in the proportions and with layer stacking probabilities found by the Fourier transform method. This reproduced the main features of the observed pattern, but a shoulder at 4·75 Å on the 5·44 Å peak was weaker on the calculated than on the observed pattern. Calculations using slightly different proportions and probabilities showed that the peak positions were slightly better represented if a smaller proportion, say 30% of 12·5 Å layers, was used. Although the two treatments are in broad agreement, there are marked differences between them; both make assumptions and there is probably considerable uncertainty in the answer that each produces. (Brown, Rayner and Weir)

The nature of illite. Illite is one of the commonest clay minerals. It occurs widely in soils and sediments and it is the most abundant source of potassium in soils. The name was first used in 1937 for fine-grained mica-like minerals that contain less potassium and more water than micas; despite continuing research, the range of chemical composition and crystal structure of illite and its mode of origin are still unresolved.

The investigation of illites from soils and sediments is hindered because they almost always occur with other clay minerals from which they cannot be separated, so that few of their properties can be measured without interference from the other minerals. To minimise this difficulty, and to study the conditions of their formation, we have synthesised illite-like minerals from mixtures of their constituent oxides.

We have studied systems containing only SiO_2, Al_2O_3, K_2O and H_2O, along the compositional boundary between muscovite $KAl_2(Si_3Al)O_{10}(OH)_2$ and pyrophyllite $Al_2Si_4O_{10}(OH)_2$, in the range 100% muscovite (100Mu) to 70% muscovite:30%

pyrophyllite (70Mu:30Py). The constituents (ammonia-peptised silica gel and solutions of aluminium and potassium nitrates) are mixed in appropriate proportions and dried and ignited at 850°C to remove ammonium nitrate. When prepared in this way the crushed and sieved gel particles superficially resemble sand grains, but they are hygroscopic, absorbing 10% water by weight, and amorphous to X-rays. Charges of about 300 mg of gel are sealed in gold tubes and heated under pressure, in Professor Velde's laboratory, for periods ranging from one day to a year, but typically for three months at 300°C and 2 kb (approximately 2000 atmospheres) pressure. The product is usually caked, but it is more porous than the starting gel material, the bulk density decreasing as the proportion of water in the mix is increased. The layer silicate product is contaminated with boehmite (γ-AlOOH) if the mix is too dry. Generally the wetter the mix the more rapidly the reaction goes to completion. The product comprises aggregates of small intergrown crystals that show no preferred orientation. The small crystals are platy for the 100Mu composition but an increasing proportion of laths are formed as the composition is altered towards that of pyrophyllite. The crystals are intergrown so that only a small proportion can be freed from the mass, even after prolonged treatment with an ultrasonic probe. It is therefore difficult to orientate the crystallites sufficiently to be able to study their basal reflections in detail by X-ray diffraction.

To understand the nature of the reaction, and to eliminate unnecessarily long periods of treatment, it is important to be able to estimate the proportion of the charge that has crystallised. Treatment with sodium hydroxide solution after grinding dissolves the gel material completely, whereas only a small proportion of crystalline material, for example 2% for the 100Mu product, is dissolved.

Previous work suggested that there are three common crystalline forms for micas of the muscovite composition; these are the 1M and 1Md one-layer monoclinic forms, both of which seem to be thermodynamically metastable with respect to the third form, the two-layer monoclinic, $2M_1$, form. So far we have not succeeded in making the 1Md form, and attempts to make it at 300°C and 2 kb have resulted in material intermediate between the 1M and 1Md forms. The 1M disorder-free form can be made at 600°C but it is rapidly converted to the $2M_1$ form at this temperature.

In the 100Mu sample approximately 10 me 100 g^{-1} of the original potassium is readily exchangeable; examination of the material by X-ray diffraction before and after cation exchange, with and without heat treatment, shows no changes in the pattern. It is concluded that most of the potassium removed by cation exchange, which represents about 4% of the total, is situated on external surfaces. As the starting material is changed towards the 70Mu : 30Py composition, i.e. to contain less potassium and aluminium, the amount of exchangeable potassium increases to about 25 me 100 g^{-1}. The change in composition is accompanied by a marked broadening and asymmetry of the mica basal reflections, a feature commonly reported for illite, and the appearance of a second layer silicate phase. This mineral swells during glycol solvation, and it is composed of illite layers interstratified with swelling beidellite. From compositions more pyrophyllite-rich than 80Mu : 20Py the illite component of the product is thus contaminated by the presence of other phases and therefore cannot be studied in detail. Within the compositional range 100Mu to 80Mu : 20Py, however, the amount of the interstratified mineral is small, and as there are marked changes in the X-ray diffraction patterns that signify the change from muscovite to illite, the nature of these illites and their relation to muscovite can be examined. (Brown, Rayner and Weir, with Professor B. Velde, University of Paris)

Clay minerals in loess. The clay minerals in $< 0{\cdot}1$ μm fractions from B and C horizons of two soils developed in loess were examined at the request of Professor U. Schwertmann of Munich. The fractions consist of dominant expanding 2 : 1 layer lattice minerals with lesser amounts of kaolinite, illite and chlorite. The interstratified minerals are complex, with components consisting of non-expanding illite interlayers, expanding interlayers, with a range of interlayer charge varying from that of low-charge smectite to high-charge vermiculite and chlorite-like hydroxy interlayers that show varying degrees of resistance to collapse when heated. These minerals, which differ slightly in the relative proportion of their components between horizons and profiles, may be somewhat inexactly described as randomly interstratified illite : intergrade smectite-vermiculite-chlorites. They quite closely resemble the clay minerals described in soils in loess from Pegwell Bay, Kent (*Rothamsted Report for 1969*, Part 1, 77–78). Because of their complexity the minerals are imperfectly understood; they are of great agricultural importance because they are constituent minerals in materials of loessial origin, which form major parent materials of many of the most valuable agricultural soils in Britain and, indeed, of the Northern Hemisphere. (Weir)

The weathering of mica in soil. The weathering of mica in soils has agricultural significance in that micas are nearly universal components of soils of the temperate regions and are a source of potentially available potassium. Further, vermiculite, which is a weathering product of mica, strongly sorbs potassium from solution, and has been implicated in the fixation of potassium in some soils. Although it is well established that mica can be altered to vermiculite-like products in laboratory experiments, such products have rarely been identified in soils, and for some time it has been suspected that different products are often formed under soil conditions. During our work on the release of potassium in British soils we found that a soil developed on Permian marl in Devon, is highly micaceous at depth but contains expanding layer silicate near the surface. The mineralogical simplicity of this parent material, which contains only illite with minor amounts of chlorite and sesquioxides, suggested that a mineralogical study of this soil would further our understanding of the pedogenic weathering of mica.

Coarse and fine clay fractions from three depths (0–10 cm, 30–42 cm and 70–78 cm) have been examined in detail by X-ray diffraction, total chemical and selective dissolution analysis, and infra-red spectrometry. As the sample from 30–42 cm showed evidence of illuvial accumulation, the main comparisons have been between the samples from 0–10 (I) and 70–78 cm depth (III). The main structural difference between these is that fine clay ($<0\cdot3$ μm) from I contains much more expanding layer silicate mineral than III, whereas III ($< 0\cdot3$ μm) contains about 80% of a mineral that shows mica-smectite interstratification with less than 20% smectite layers. I ($<0\cdot3$ μm) contains about the same amount of a mineral with approximately 60 : 40 mica-smectite layers, with evidence for small amounts of a vermiculite or aluminium-interlayered component. After subtracting the dithionite-extractable Al and Fe, the atomic proportions of the two calcium-saturated clays, per $O_{20}(OH_4)$, are:

	Si	Al	Fe	Mg	K	Na	Ca
I ($< 0\cdot3$ μm)	6·82	4·29	0·54	0·47	0·77	0·08	0·22
III ($< 0\cdot3$ μm)	6·93	4·01	0·50	0·66	1·10	0·08	0·13

The main differences between the samples is the decrease in the (Al + Fe) : Si ratio with depth, the increase in the Mg and K contents, and the decreased amount of Ca. As the change in the Ca content does not balance that of K, in terms of charge, the surface clay has a smaller charge density than the deep clay.

Interpretation of these differences in terms of a pedochemical alteration of the deep clay assumes an initial uniformity of soil-forming material, an assumption that is frequently not valid in English soils where surface addition occurs by transportation.

However, the fine clay fractions of the surface and deeper horizons of this soil do not contain kaolinite, and as this is an almost universal constituent of soils, it is unlikely that the surface has been contaminated by transported material.

The differences between I and III are therefore interpreted as the result of pedochemical weathering of illite, in which by the removal of Si, Mg and K the micaceous clay is transformed mainly into a mixed-layer mica-smectite, with relatively smaller amounts of vermiculite or interlayered vermiculite-smectite. Such a transformation explains the relative infrequency of vermiculite and the phenomenon of potassium fixation in soils. (Branson and Newman)

Clay minerals in the sediments of Lake Abert, south-central Oregon, USA. The results of an extensive study of the hydrology and chemistry of Lake Abert (Phillips, K. N. & Van Denburgh, A. S. *USGS Professional Paper* (1971) 502-B) showed that much of the dissolved potassium, magnesium and silicon brought into the lake is lost to the lake-bottom sediments. It was originally thought that all the lake sediments had formed by precipitation from solution, and the study was conceived as one dealing with authigenic clay mineral formation under hypersaline conditions. It is now clear that the majority of the clay minerals in the lake originated as weathering products of the surrounding igneous rocks and were transported to the lake by soil creep or in flash floods. One component has been identified as coming from an extensively weathered dacitic pumice tuff that outcrops on a steep cliff face on the eastern edge of the lake. The clay mineral in this is a smectite-rich randomly interstratified smectite-illite-chlorite. However, the outcrop does not seem to be sufficiently extensive to account for all the lake sediments, and further sampling is being undertaken to find a clay weathering product of the basalt that forms the major part of the cliff. Only when the full range of clay inputs to the lake is known will it be possible to deduce the extent of authigenic clay mineral formation. The lake clays contain more K, Mg and Si than the pumice tuff clay; in fact they contain so much silica that the composition cannot be recalculated in the form of a 2 : 1 layer silicate mineral. The diffraction patterns of the lake clay have proved difficult to interpret. Superficially they resemble those of randomly interstratified smectite-illite-chlorites with greater proportions of illite and chlorite layers than the pumice tuff clay, but their proportions are not consistent with those of a simple smectite-chlorite-illite interstratification and, as in the loess soils described above, the presence of intergrades is suspected. (Weir, with Dr. Blair F. Jones, US Geological Survey)

Mineralogy of Nigerian soils. The study of Nigerian soils has continued, using the techniques described in last year's Report. We have examined some 200 samples, covering 40 soil profiles from 11 toposequences; the toposequences represent the six main climatic zones of Nigeria and the soils overlie granites and gneisses, amphibolites, sedimentary formations, quartzites and quartz-mica schists.

The general impression is one of uniformity of the clay fraction. Minerals of the kaolin group, with subsidiary iron oxides, predominate in the majority of soils; other minerals become important where the parent material or the topography permit. Micas are common in the clays from profiles developed over mica-rich rocks such as schists and micaceous gneisses; expanding layer silicates are major components in four valley site profiles, and in one profile on amphibolite in the mid-slope position. Two types of mica, probably muscovite- and biotite-like, have been recognised in many of the mica-containing soils. Interstratified 2 : 1 layer silicates occur in many profiles, but these are never dominant.

The kaolin group minerals are of two distinct types: a kaolinite-like mineral, with affinities to b-axis disordered kaolinite, and a tubular halloysite-like material. The former occurs in every sample; the latter is found in small amounts in many profiles and is common in the profiles developed on amphibolite and in the two toposequences on Intermediate Crystalline rocks in the wettest climatic zone. It is least abundant in soils developed in the drier regions. In all of these the halloysite occurs as short tubes or rolled plates approximately $< 0{\cdot}3$ by $< 0{\cdot}05$ μm. In the upslope member of the Ibadan toposequence (Ekiti series) the halloysite is in the unusual form of very long tubes up to 6 μm long by 0·04 μm diameter.

The iron oxides that have been recognised are goethite, hematite and rarely lepidocrocite. Goethite is present in all the soils except for valley site profiles. Hematite is less widespread and is generally most common in upslope and midslope profiles, decreasing in amount downslope until, in most of the valley sites, it is absent.

The only aluminium oxide is gibbsite, which occurs quite commonly across the whole range of parent materials. It is most common in the high rainfall areas and in the upper soil horizons, where it probably represents the residue of strongly weathered materials. However, its presence in two lithosols suggests that if conditions are suitable it may also be an early weathering product. The amount of the total oxides rarely exceeds 15%, with gibbsite usually $< 5\%$ and iron oxides 5–10%.

Other minerals present in the clay fractions, all $< 10\%$, are quartz and potassium and sodium feldspars. Quartz occurs in almost all the soil clays, but it is rare or absent in soils over quartzite. On the same or similar parent rocks it tends to decrease in amount towards the wetter regions. The occurrence of feldspars is variable and reflects their abundance in the parent material.

The fine sand mineralogy generally reflects the constitution of the parent rocks, with quartz being dominant over varying amounts of micas, feldspars and more resistant minerals. Rarely other minerals such as hornblende and garnet are important. The feldspars are usually alkali feldspars, although plagioclase feldspars do occur, especially on intermediate crystalline rocks. In general there is a tendency for quartz in the fine sand to be more common in upper horizons and down-slope. Some profiles that have uniform clay mineral assemblages throughout show abrupt changes in their fine sand mineralogy.

The preliminary analyses showed that the Ekiti series soil from Ibadan contained very long tubes of halloysite, and a more detailed study was made to find the source of this unusual material. The < 2 μm fraction separated from the weathering parent rock (biotite-gneiss) consisted largely of this halloysite mineral. When pieces of orthoclase and biotite from the weathering rock were examined by scanning electron microscopy the surface of the feldspar was seen to consist of fibrous material which appears in the clay fraction as the long tubular particles of halloysite.

The X-ray diffraction pattern from oriented films of the < 2 μm fraction of the same weathered rock gave a strong reflection with a spacing of 4·86 Å. The strongest reflection of gibbsite has a spacing close to this value, but as the reflection persisted after heating to 335°C, it must have derived from something other than gibbsite. After concentration by removing HF-soluble matter, the material responsible for the 4·86 Å reflection was identified as bastnaesite, which is a rare earth fluoride carbonate (La, Ce, etc.) $(CO_3)F$. Comparison of the X-ray diffraction patterns of the original clay fraction and the HF-insoluble residue of the whole soil showed that the bastnaesite was not an artefact formed by HF in the concentration process. Eleven rare earth elements have been identified by X-ray fluorescence spectrography in the concentrate and in the < 2 μm fraction of the weathered rock. In decreasing order of abundance the rare earths found are La, Nd, Ce Pr, Sm, Gd, Dy, Eu, Er, Tb and Ho; Y and Th and also Ca are present in appreciable amounts. Rare earth elements, in lesser abundance, were also identified in the clay fractions of soils overlying this rock but neither bastnaesite nor any other rare earth mineral could be identified in the residues obtained after treating the clay fractions with HF. (Brown and Hughes)

Mineralogy of Solomon Island soils. The fine sand (60–250 μm) and clay (< 2 μm) fractions of 131 samples of subsurface (B) and subsoil (B or C) horizons from 63 profiles representing the main soil groups in the Solomon Islands were analysed mineralogically to assist part of the concluding work of the Ministry of Overseas Development (Land Resources Division) in this part of Melanesia. The purpose of the work is to determine soil parent materials and weathering processes more precisely than had been possible in the field, to help interpret soil chemical data, and to clarify the status of some of the soils in the American system of soil classification used by Land Resources Division.

When necessary the air-dry samples were decalcified with acetic acid buffered at pH 5, and then dispersed by shaking and brief ultrasonic treatment in water made alkaline (pH 10) by the addition of sodium carbonate. Some of the soils, classified as Andepts, could be dispersed only by repeated applications of 0·0002N hydrochloric acid (pH 3·25). The clay fractions were separated by repeated centrifugation and analysed by X-ray diffractometry and electron microscopy; the fine sands were separated by sieving and analysed with a petrological microscope.

On the basis of field profile morphology and selected chemical data, the soils were classified as six Eutropepts, five Dystropepts, five Ustropepts, one Humitropept, three Troporthents, three Tropohumults (one typic, one oxic, one aquic), three Hydrandepts, two Vitrandepts, ten Eutrorthox, eleven Haplorthox, two Acrorthox, three typic Tropudalfs, four oxic Tropudalfs, four Haplustox, and an Ochraquox. However, on the basis of the mineralogical composition, principally of the clay fractions, the soils could be divided into seven groups.

Group 1. Two of the Troporthents are sandy soils composed mainly of coral beach detritus, with $< 10\%$ non-calcareous material of variable mineralogy and origin. They are slightly alkaline, with little evidence for weathering in the profile, though some constituents of the clay and fine sand fractions were probably weathered before incorporation in the beach sands.

Group 2. The Hydrandepts and Vitrandepts are neutral to slightly acid loams and sands, with clay fractions composed mainly of imogolite and/or allophane, and fine sand composed mainly of basaltic glass in the Vitrandepts and of finely crystalline ash or lava fragments in the Hydrandepts. The sand fractions of the Vitrandepts are unchanged by weathering, and the clay fractions contain only small allophane spheres and feldspar. However, the Hydrandepts are more weathered; the rock fragments in their sand fractions have reddish-brown ferruginous coatings or are partly altered to gibbsite aggregates, and their clay fractions contain fibrous imogolite, gibbsite, and occasionally spheroidal halloysite, as well as allophane. The differences between the relative proportions of the main clay minerals in the subsoil and subsurface horizons of the Hydrandepts suggest that allophane is being changed to imogolite and/or gibbsite.

Group 3. All the Ustropepts and typic Tropudalfs, one Dystropept, the typic Tropohumult, the remaining Troporthent, and all but one of the Eutropepts are neutral or slightly acid loams and clays, composed mainly of 2 : 1 layer silicate minerals with subsidiary kandite. Traces of many other minerals, including quartz, feldspar, cristobalite, anatase and amphibole, occur in the clay fractions, and the fine sands also contain a wide range of minerals; the soil parent materials can be inferred thereby, even though there has been more weathering than in Groups 1 and 2. They include acid, intermediate, and basic or ultrabasic igneous rocks, alluvium derived largely from volcanic rocks, and glauconitic marine sediments.

Group 4. The Humitropept and most of the Dystropepts are slightly acid clays containing kandites, goethite, and less 2 : 1 layer silicates than the soils of Group 3. In both groups, the 2 : 1 minerals are mainly interstratified types, with variable amounts of vermiculite- and smectite-like layers. The fine sands of the Group 4 soils contain mainly iron oxide aggregates, and weathering has removed most of the evidence for the nature of the parent materials.

Group 5. All the Haplustox and oxic Tropudalfs, most of the Eutrorthox and Haplorthox profiles, and the single Ochraquox, oxic Tropohumult and aquic Tropohumult profiles, are slightly acid clays, composed mainly of kandites, with subsidiary crystalline hydrated oxides and little or no 2 : 1 minerals. The kandites correspond partly to kaolinite and partly to halloysite, and commonly form curled plates, short tubes or spheroidal particles. The oxides are mainly goethite and gibbsite, but boehmite and nordstrandite also occur in the Eutrorthox profiles over limestones. The small amounts of fine sand contain mainly iron oxide aggregates, quartz and primary iron ores (magnetite and ilmenite), with some gibbsite aggregates in the Eutrorthox, Haplorthox and oxic Tropohumult profiles. This assemblage results from strong weathering, which has destroyed most of the evidence for the parent materials. Small amounts of fresh volcanic material, usually andesitic, occur in many of the soils, but are more abundant near the surface than in the subsoil, and must have been deposited as ash from the active volcanoes in the Solomon Islands quite recently, at least after most of the weathering in these profiles had occurred.

Group 6. The Acrorthox, one Eutropept, four of the Haplorthox and three of the Eutrorthox profiles are slightly acid to slightly alkaline clays or fine loams, composed almost entirely of crystalline hydrated oxides; these are mainly iron oxides (goethite and haematite) in the Acrorthox and Haplorthox profiles, which overlie ultrabasic igneous rocks, but mainly aluminium oxides (gibbsite with subsidiary boehmite and nordstrandite) in the Eutropept and Eutrorthox profiles, which are on coral limestones. The Eutropept and Eutrorthox also contain a little crandallite (calcium aluminium phosphate) in their clay fractions. The fine sand of soils in this group are also composed mainly of iron and aluminium oxides, and some contain small amounts of unweathered recent volcanic ash.

The Eutrorthox soils in this group are sufficiently rich in residual aluminium oxide to be potential sources of mineable bauxite, but the source of the aluminium is puzzling, as the underlying limestones are unlikely to contain enough aluminous material to build up the residual bauxite profiles, even over a very long period of weathering. However, the presence of volcanic ash suggests that the aluminium is the residue from weathering of successive ash falls over a long period; this is true even for bauxite deposits on atolls, such as Rennell Island, previously thought to be too remote (> 200 km) from the nearest volcanoes.

Group 7. This includes one profile only, a Eutrorthox from Bellona atoll, composed almost entirely of crandallite, with a little goethite in the clay fraction, and traces of volcanic ash in the fine sand. As in the bauxitic profiles, the aluminium necessary for the formation of crandallite must have been derived by long continued weathering of successive falls of far-travelled ash. Similar phosphatic soils over limestone occur on other Pacific atolls and in parts of New Guinea, and the phosphorus in these is attributed either to guano or to residual accumulation from the limestone beneath. Whatever its origin, the phosphorus is fixed in a very resistant mineral, which seems to be one of the final residual products of prolonged severe weathering.

This grouping of the profiles reflects mainly the extent of weathering, but also the nature of the soil parent materials, especially in the less strongly weathered soils (Groups 1 and 2). The degree of weathering varies widely, the range including no recognisable alteration in calcareous sands (Group 1), the formation of small amounts of amorphous or poorly crystalline clay from volcanic materials (Group 2), the development of 2 : 1 layer silicates from various parent materials (Group 3), the progressive replacement of these by kandites and hydrated iron oxides (Groups 4 and 5), and the progressive replacement of kandites by aluminium oxides (Groups 5 and 6) or (where phosphate was available) by crandallite (Groups 6 and 7). Most of the soils have little or no potassium-containing minerals, and the natural fertility, particularly of the more strongly weathered profiles in Groups 5–7, depends upon periodic rejuvenation by ash showers. (Brown, Catt and Ormerod)

Relationship between moist and dry colours of boulder clay soils. The moist colours of soils are recorded in routine descriptions by the Soil Survey and others, but the causes of many colour differences are still imperfectly understood. During our work on the weathering of boulder clays in eastern England, we recorded the colour of samples after air-drying as well as the more usual moist field colours, and the two sets showed a consistent relationship that seems pedologically significant. For samples of boulder clay taken below approximately 5 m depth, the air-dry colours were consistently lighter in value than the moist colours, by two units on the Munsell Color Chart; for example, the Devensian Drab Till is a uniform very dark greyish brown (10YR 3/2) when moist and greyish brown (10YR 5/2) when air-dry, and the overlying Purple Till is dark brown (7·5YR 3/2) moist and brown (7·5YR 5/2) dry. In contrast, samples from nearer the surface, which are brown (7·5YR 4/2 to 7·5YR 4/4), reddish brown (5YR 4/3 or 5YR 4/4), or dark reddish brown (5YR 3/4) when moist, changed colour on drying in various ways; they became lighter in value by 1, 2 or 3 units, many changed in chroma, and some also in hue.

The till at the surface was originally thought to be a separate depositional unit (the Hessle Till), but our mineralogical work showed that it is an oxidised form of either the Drab or the Purple Till (*Rothamsted Report for 1974*, Part 1, 194–195). The oxidative weathering has not only changed the moist till colours, making them redder in hue, lighter in value and brighter in chroma, but has also caused the tills to change colour less predictably on drying. The reason for this is obscure, but is probably related to changes in the nature and amount of hydrated iron oxides, resulting partly from the oxidation of sand- and silt-sized pyrites and siderite grains, and partly from reduction and re-oxidation of iron with fluctuating groundwater drainage conditions. The relationship between air-dry and moist colours needs to be observed in other soils and parent materials, but it is possible that a simple test can be devised on this basis to distinguish between recently oxidised and completely unaltered parent materials in deep subsoil horizons. (Catt)

Applications of infra-red spectrophotometry to soil investigations. We are studying the possible uses of infra-red techniques in soil mineralogy. Two types of application have been evaluated: the qualitative correlation of infra-red bands with structural classes of clay and other minerals, and the semiquantitative determination of selected minerals in soil clays. In qualitative mineralogy, infra-red spectrophotometry is sensitive enough to detect amounts of certain minerals (e.g. kaolinite and chlorite) too small to be detected by X-ray diffraction. It is also possible to make inferences about the composition of clays, for instance the composition of the octahedral layers in layer silicates. The technique can also be used to detect the presence of substances sorbed on clays, for example organic compounds and phosphates.

The main problem associated with the quantitative analysis of minerals by infra-red spectrophotometry is the preparation of suitable standards. The application to mineral determination has been concentrated on the compensation method which involves the introduction of the standard into the reference beam, to balance absorption due to the component whose concentration is required. This technique has given good results for quartz and kaolinite, and it is much faster than selective dissolution methods; it is more accurate for kaolinite than methods based on X-ray diffraction. (Branson)

Clay physics

Soil structure. As part of the programme of the Working Group on Soil Structure we are comparing the pore structure of two pairs of soils, from the Denchworth and Evesham, and the Hanslope and Ragdale series, with the object of testing the accuracy of our techniques, and to construct a model for describing the changes of the pore structure of a clay soil with changing moisture content. Should there be significant differences between the pairs of soil that we have chosen, we shall attempt to relate these differences to the compositions of the soils and to the treatments to which they have been subjected, details of which will become available from other contributors to the project.

Field-moist aggregates have been dried by solvent replacement and by air-drying, and we are attempting to determine the pore size distribution, at least for the coarser pores, of replicate samples by high pressure mercury intrusion and by organic liquid retention. The mercury intrusion measurements are done commercially, and we are examining the intruded samples to see whether artefacts are produced by this method; preliminary results indicate that this does not happen.

Further replicate samples are being dried by equilibration on suction plates, and on pressure membrane and isopiestic apparatus; the consequent shrinkage is being measured volumetrically by displacement after coating the aggregates with resin.

The results obtained thus far on the Denchworth and Evesham samples show that the aggregates shrink normally at first, i.e. the volumetric shrinkage equals the decrease in the gravimetric water content, but that air penetrates the sample once the water content falls below about 20%. These results illustrate that the pore structures of air-dried clay soils are very different from those of field moist material. (Newman and Perrins)

Soil geochemistry

Minor elements in sedimentary parent materials. Large proportions of the potentially available minor elements in aerobic sedimentary deposits occur in sesquioxide weathering products, rather than in the silicate clay minerals or the weathered sand and silt minerals. The technique of extracting this sesquioxide fraction with acid ammonium oxalate in UV light (Le Riche, *Geoderma* (1973) **9,** 43–57) was used in a modified form to compare the minor element assemblages in samples of two sedimentary formations: Upper Greensand (Cretaceous) and Thanet Beds (Paleocene).

Materials ground to 250 μm were first decalcified with acetic acid at pH $\nless 4$ and the residues extracted with acidified H_2O_2 to test for peroxide-soluble Mn minerals, the minor element contents of both solutions being measured. The residues from these treatments were treated under UV light with ammonium oxalate to extract the sesquioxides and associated minor elements, and the extracts dried and ignited.

To avoid spurious correlations resulting from the diluting effect of varying amounts of inert mineral material and calcium carbonate, the quantitative relationships between the extracted elements were considered as the concentrations in these ignited extracts rather than in the original materials.

There was little organic C in either group (mostly $< 0{\cdot}5\%$) and only in the Upper Greensand group was there any correlation between organic C and any of the elements measured (Fe_2O_3, SiO_2, Cu, Mn; $r > 0{\cdot}6$). In this more strongly calcareous group, extractable Mn was correlated strongly, but Sr only weakly, with the $CaCO_3$ in the original material (Mn, $r = 0{\cdot}94$; Sr, $r = 0{\cdot}22$), most of these two elements being extracted during decalcification; but in the weakly calcareous Thanet Beds group, Sr was and Mn was not correlated with $CaCO_3$ (Mn, $r = -0{\cdot}28$; Sr, $r = 0{\cdot}81$). In the extracts from the Upper Greensand group Mn was correlated only with Fe ($r = 0{\cdot}82$) whereas in the Thanet Beds it was strongly correlated with Co ($r = 0{\cdot}93$), and weakly with Al, Ni, SiO_2 and V. Only small proportions of the associated Mn and Co were dissolved by peroxide, in contrast to the findings of Taylor and McKenzie (*Australian Journal of Soil Research* (1964), **2,** 235–248) with Australian soils.

The concentrations of Cr and Ti were correlated in the original materials of both groups, and although only about 10% of the Cr and 3% of the Ti were extractable, the amounts in the ignited extracts, were also correlated. This is consistent with the bulk of both elements being associated together in resistant minerals and with their behaving similarly in the weathering products. V behaved similarly to Cr and Ti in the Thanet Beds, but was unrelated to them in the Upper Greensand, in which it was most closely associated with Cu and Ni.

Co, Cu, Ni, V and Zn were more closely correlated in the extracts of the Upper Greensand than of the Thanet Beds but in the latter group Co and V were barely related to the others because of their closer respective relationships with Mn and Cr.

It is evident that there are differences, that could be inherited by soils developed on them, between the associations of minor elements in these two formations. (Le Riche)

Geochemistry of the soils of Pembrokeshire. The interpretation of data on 13 minor elements in the soils of Pembrokeshire has continued, the survey now incorporating S.W. Pembrokeshire as well as the northern area discussed in *Rothamsted Report for 1972*, Part 1, 75. Correlation matrices have been computed for the N. Pembrokeshire data, dividing the soils on the basis of drainage and rock type. For example, correlation matrices for soils developed on three different groups of rocks show very varied patterns. In soils of the Ordovician sediments, Br and Pb are strongly related to organic matter in the topsoils and Zn, Cu, Ni, Fe and Mn are all very closely related, as are Sr, Y and Rb. ($P = 0{\cdot}01$.) In the rhyolite soils, Br is still correlated with organic matter but Pb has more affinity with Zn, and there are fewer correlations. The soils of the intrusive igneous rocks (diorites and dolerites) have many strongly correlated elements, with Fe, Mn, Pb, As, Zn, Cu and Ni forming a group. In these soils, Br is related to Y in both top and subsoils. Samples taken at 10 cm depth from grid square SM93 were analysed and correlation matrices are being computed to show the relationships between major and minor elements in the soils of various soil types and rock types in this square. (Devonshire, Perrins and Williams, with Rudeforth, Soil Survey)

Distribution of Br in soils and herbage of N.W. Pembrokeshire. Little is known about the occurrence of Br in soils and plants. Bromine is not thought to be an essential element for plant growth, and until recently it was considered unimportant; however, with the inclusion of Br in components of petrol, and the use of methyl bromide as a soil fumigant, it would be useful to assess the range of the Br contents of uncontaminated agricultural soils and herbage.

Br has been determined in the soils and herbage of N. Pembrokeshire by a semi-quantitative X-ray fluorescence spectrometric method. The mean amounts and ranges of Br in the soils were 53 ppm (10–420 ppm) in topsoils and 48 ppm (5–455 ppm) in subsoils. The average herbage concentration was 45 ppm and the range 5–157 ppm (dry basis).

The topsoil Br content was correlated with the loss on ignition, but correlations were better if soils of each drainage class were plotted separately, thereby showing a three-way relationship between drainage, organic matter and Br. The Br contents of topsoils that were larger than those predicted by the Br-loss on ignition regression equation were thought to be due to contamination, but the pattern is confused because of the very great maritime influence.

The distribution of Br down the profile varies with drainage status. The freely drained soils have similar amounts of Br in the subsoil (50 cm) and the topsoil, but the more poorly drained soils have less Br in the subsoil.

There is no clear relationship between the contents of Br in the herbage and the soil, probably because of differential contamination of soils and herbage under the maritime influence to which the area is subject. (Williams)

The distribution of Pb in the soils and herbage of Pembrokeshire. Pollution by Pb near main roads and areas of heavy population is an increasing problem. It is difficult to assess the extent of pollution of soils and herbage until the range of concentrations of the element in unpolluted areas is known. A study of the Pb concentrations in soils and herbage of Pembrokeshire, which is an agricultural area in which industry is almost wholly confined to the south, has given an indication of the amounts of Pb that might be considered as 'background' concentrations.

In Pembrokeshire the soil Pb concentrations are very variable; topsoils contain 2–428 ppm (mean 40 ppm), and areas around Milford Haven, Haverfordwest and Pembroke Dock are characterised by large concentrations of Pb in the topsoil. We have determined the Pb contents of the herbage from NW Pembrokeshire. Almost half the herbage samples contained less than 1 ppm Pb, on a dry basis; the maximum was 14 ppm. The Pb content of the herbage was subject to seasonal variations, samples harvested in the spring containing more Pb than material collected later in the year. In part, this is likely to be due to soil contamination. (Williams)

Geochemistry of reclaimed sediments of the Wash. An investigation of the geochemistry of reclaimed sediments from the Wash has been started. In the area around Friskney, Lincolnshire (TF 45), marine sediments have been reclaimed for agricultural purposes from Roman times. We intend to study the geochemical changes caused by draining, and also the changes in the availability of major and minor elements for herbage. (Williams, with Robson, Soil Survey)

Soil chemistry

The effects of cropping and management on the organic matter and nutrient reserves of tropical soils. The major investigation was on the mulching experiment of the IITA Farming Systems Programme. The proportion of labile nitrogen was greater in soils under organic mulches than in unmulched soil, and the microbial biomass was also greater in organically mulched soils than in unmulched soil, or in soils under plastic mulches. There was clear evidence that the organic mulches immobilised inorganic nitrogen, immobilisation being greatest with the more woody materials. The results were influenced by soil variation across the experimental area, and a statistical treatment was suggested to allow for this in subsequent work on the experiment.

The effect of the insecticide DDT on tropical agricultural systems is being studied at IITA by staff of the Centre for Overseas Pest Research (COPR). Soil samples were taken from a COPR experiment on the effect of DDT on the yield of cowpeas. When bush was cleared and cropped to cowpeas for two years, the soil organic matter content declined by 25%. The decline in the biomass was even more rapid (50%), in agreement with our earlier work. DDT had no significant effect on the organic matter content, nitrogen mineralisation or microbial biomass carbon in cultivated plots.

The nitrogen reserves and biomasses were also measured in soil samples from the

COPR Termite Ecology project at Mokwa, Northwestern State, in the savanna zone. Although the soils contained less organic matter than soils under secondary regrowth at IITA, the results were similar, the total C and N contents declining more slowly than the biomass C on cultivation. The cultivated soils not only contained less total nitrogen than secondary woodland savanna soil, but less of the cultivated soil N was readily mineralisable.

A term 'PSN' (Percent Stress-labile Nitrogen = N mineralised after fumigation/total N in soil × 100) has been introduced as a measure of the 'quality' of soil nitrogen; it is consistently greater in bush soils than in cultivated soils and sensitively reflects changes in soil management. (Jenkinson and Tuckwell, with Dr. A. Ayanaba, International Institute of Tropical Agriculture)

The effects of air-drying on metabolism in soil. Work was continued (*Rothamsted Report for 1974*, Part 1, p. 197) on the origins of the flush of decomposition that occurs when soils are dried and rewetted, using soils from the IITA. Broadly, the 1975 results confirm and extend the 1974 findings; part of the flush caused by air-drying comes from the decomposition of those organisms killed during drying and part from organic substrates that are inaccessible to microbial attack in moist soil but are exposed when soil is dried and rewetted. To test the second part of this hypothesis, silica gel was moistened with 1% glucose and leached with water until glucose could no longer be detected in the leachate. After air-drying and remoistening, glucose was again present in the leachate, presumably having been released by syneresis during drying. A similar mechanism could well operate in soil. (Jenkinson and Tuckwell)

The accumulation of organic matter in soils from the Hoosfield Permanent Barley experiment. Soil samples taken from the experiment at intervals since 1882 were reanalysed by modern methods, together with a set of samples taken in 1975. The soil receiving farmyard manure annually is still accumulating organic matter, and equilibrium has not been reached after 124 years; the turnover time of the organic carbon in this plot is about 30 years. These results will be used in an attempt to develop a more accurate model of the turnover of organic matter in soil than that currently in use. (Jenkinson)

Reactions between minor elements and organic matter. We have continued our study of the non-colloidal organic metal complexing agents formed by decomposing plant matter (*Rothamsted Report for 1972*, Part 1, 76). Similar organic compounds have been found in the decomposition products of aerobically and anaerobically incubated plant matter, in the solution expressed from peat, in the dialysable fractions of alkali extracts of soil and peat, and in soil solutions from a calluna podzol. Whereas the non-colloidal fraction of the products of aerobically decomposed plant matter (*Rothamsted Report for 1972*, Part 1, 76) forms complexes with Cu, Ni, Co, Mn, Zn and Cd, only Cu is unquestionably complexed by the corresponding anaerobic decomposition products.

Ultrafiltration of the various solutions showed that the complexing activity derives from several compounds, the molecular weights of which range, very roughly, from < 1000 to some tens of thousands. The < 1000 mol. wt. material is responsible for a considerable fraction of the total complexing capacity of the extracts, and in terms of the amount of Cu complexed per atom of C, this fraction is more active than the larger molecular weight material. The complexing activity resides in the acidic fractions of the various extracts.

The specific activities of water extracts of composted lucerne or peat are increased by contact with KOH, so that the complexing activity of alkaline soil extracts is in part caused by artefacts. The production of complex-forming artefacts is decreased by excluding atmospheric oxygen from the alkaline extracts, but it is not entirely prevented.

Several complex-forming constituents of the various extracts have been identified by gas–liquid chromatography, but the concentrations of these compounds (succinic, p-hydroxybenzoic, vanillic, syringic, p-coumaric and, possibly, malic acids), and the stabilities of their Cu complexes, are too small to account for the properties of our extracts. (Bloomfield and Sanders)

Heavy metals in sewage sludge. Continuing our work on the behaviour of heavy metals in contaminated sewage sludge (*Rothamsted Report for 1974*, Part 1, 198), we find that the water solubilities of the metals are affected by aerobic incubation to very different extents. After eight months' incubation, respectively 26, 12 and 6% of the total Ni, Zn and Cd were extracted by water, compared with only 1–2% of the total Cu, Pb and Cr.

After removing colloidal particles from the extract by filtering through a 0·45 μm filter, the concentrations of the metals were not affected by subsequent removal, by filtration, of material of mol. wt. $>$ 1000, so that quite small molecular species are involved.

The extents to which the dissolved metals were retained by cation and anion exchange resins indicates the probability that Zn, Pb and Ni were in cationic forms, whereas Cu, Cr and Cd behaved ambivalently, as would be expected if they were combined in dissociable anionic complexes. Preliminary results obtained with a Cu-electrode show that half the water-soluble Cu is in complex combination.

An appreciable concentration of the organic fraction of the sludge was obtained by centrifuging after ultrasonic dispersion and decantation. The mineral residue contained only 2–3% of the original total metal content of the sludge, and significant amounts of only Ni and Zn were lost in water-soluble forms.

Despite its relatively large content of heavy metals, the sorptive capacity of the organic concentrate from the sludge was far from saturated—after treatment with excesses of the individual metals the oven-dry products contained 3·4% Pb, 2·1% Cu, 1·8% Hg and around 1% of Ni, Zn, Cr and Cd. (Bloomfield and Pruden)

The fixation of Cu by peat. In collaboration with Dr. Ng Siew Kee (United Plantations BHD, Malaya) we recently started an investigation of the fixation of Cu by peat. Samples of virgin peat, and peat from an oil palm plantation, contained around 10 ppm total Cu. In an attempt to correct Cu-deficiency, an area had been treated with 2·5 kg $CuSO_4 . 5H_2O$ per palm tree. We found that this Cu had been almost completely retained in the top few cm of the soil, the Cu content of the 8–20 cm zone being only some 10% of that of the top 8 cm. This result suggests that it might be more effective to apply Cu dressings to the palms through auger holes bored to the depth of the rooting zone.

The Cu-fixing properties of the Malayan peat were very similar to those of an acid lowland peat bog from N. Wales, with which it was compared. The amounts of Cu sorbed from $CuSO_4$ and retained after exhaustive dialysis were greatest between pH 5–7, and within this range were not greatly affected by the pH. The Cu-fixing capacities of the peats were significantly smaller at pH 4, and they decreased sharply under conditions more acid than this.

Weight for weight, the Cu sorption–desorption characteristics of the original peats and the corresponding NaOH-extracted fibrous matter were almost identical. Making the probably unjustified assumption that the effects are additive, the fibrous fractions were responsible for roughly half the total Cu-fixing capacities of the original peats, so that it seems that the unavailability of Cu in such material is not exclusively caused by what is commonly considered as humidified organic matter. We hope to continue this study. (Bloomfield and Pruden)

Staff and visiting workers

Dr. V. N. Kudeyarov, of The Institute of Agrochemistry and Soil Sciences of the USSR Academy of Science, worked with us for two months on the effects of CS_2 on soil metabolism. Dr. Blair F. Jones of the US Geological Survey spent some weeks in the department, continuing his study of the clay minerals of Lake Abert, Oregon; Dr. R. J. Gilkes arrived in August to spend a year studying the mineralogy of micronutrient cations in soil.

D. S. Jenkinson read a paper at the Welsh Soils Discussion Group Meeting on Soil Organic Matter, and J. R. Sanders gave a talk on 'Interactions of trace metals with Soil Organic Matter' at The University of Hull. J. A. Catt spoke at the conference of The Institute of British Geographers at Oxford on 'Soils and geomorphology of the English chalk landscape', at a meeting of the Geographical Association on 'Geomorphology, Soils and Agriculture', and at the Council for British Archaeology conference at Reading on the landscape of the lowland zone in Britain. Catt also organised a conference of the Quaternary Research Association at Manchester on Quaternary Climates, and helped with the organisation of a joint meeting of the Quaternary Research Association, the Challenger Society and the Marine Studies Group of the Geological Society, on the Quaternary history of the continental shelf around Britain. C. Bloomfield lectured at the Louisiana and Ohio State Universities, USA, and spoke at a meeting held in York to discuss the reclamation of pyritic mine waste.

Publications

GENERAL PAPERS

1 POWLSON, D. S. (1975) Effects of biocidal treatments on soil organisms. In: *Soil Microbiology*. Ed. N. Walker. London: Butterworths, pp. 193–224.

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2 BLOOMFIELD, C., KELSO, W. I. & PRUDEN, G. (1975) A comparison of the compositions and properties of natural and laboratory-prepared humified organic matter. *Soil Biology and Biochemistry* **7**, 313–317.

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4 GALLAVAN, R. C. & GREENE-KELLY, R. (1974) The effect of evaporation of entrained liquid on soil fabric. *Journal of Soil Science* **25**, 499–505.

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