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Rothamsted Experimental Station Report for 1965

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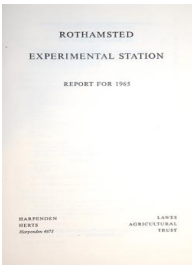

Pedology

C. Bloomfield

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PEDOLOGY DEPARTMENT

C. BLOOMFIELD

G. Brown visited Sweden at the invitation of the Swedish Clay Minerals Society and contributed to the Society's spring meeting in Stockholm; he also lectured to the Mineralogical Institute at Uppsala. R. Greene-Kelly undertook the editorship of *Clay Minerals*. Ingr. R. Gabriels, of the Rijkslandbouwhogeschool, Ghent, spent a month in the department studying spectrographic techniques. Professor F. L. Himes, of the Ohio State University, arrived in December to spend six months studying metal–soil-organic matter interaction.

The Pedology Department's concern is the study of the fundamental properties of soil—its genesis and composition, and its static and dynamic behaviour. Our effort is divided between the study of the mineralogical and chemical composition of soils, clay mineralogy, the weathering of soil minerals, the physical properties of soil in relation to its composition, the dynamics of soil organic matter and the role of organic matter as an agent of soil-forming processes.

Mineralogy

Structure of pyrophyllite and talc. The study of the structures of these uncharged layer silicates was continued. Small crystals cut from a clear cleavage sheet of talc, obtained from Dr. Sterling B. Hendricks, were examined; the sample came from Harford County, Maryland, where Hendricks obtained the sample used in his earlier study (*Z. Kristallogr. Kristallgeom.* (1938) **99**, 264–274). The diffraction patterns, taken for rotations about the three distinct 5·3 Å axes, show that the crystals are triclinic, with a primitive cell $a = 5{\cdot}29$, $b = 5{\cdot}30$, $c = 9{\cdot}47$ Å, $\alpha = 86{\cdot}1°$, $\beta = 98{\cdot}9°$, $\gamma = 120{\cdot}0°$, and not monoclinic as Hendricks reported for his sample. A *C* face-centred cell can be chosen to describe the triclinic sample $a = 5{\cdot}29$, $b = 9{\cdot}18$, $c = 9{\cdot}46$ Å, $\alpha = 90{\cdot}6°$, $\beta = 98{\cdot}9°$, $\gamma = 90{\cdot}0°$, which shows the close similarity to Hendrick's monoclinic cell $a = 5{\cdot}27$, $b = 9{\cdot}13$, $c = 18{\cdot}88$ Å, $\beta = 100{\cdot}2°$ ($\alpha = \gamma = 90°$ exactly). α is significantly greater than 90° with our sample, and the intensities do not show monoclinic symmetry. The samples are thus clearly different; they probably consist of essentially similar layers that are more regularly stacked in our sample.

The diffracted spots from talc are much sharper than from pyrophyllite, talc spots with indices with $k \neq 3n$ being nearly as sharp as spots with $k = 3n$. Disorder is shown only by weak streaks along c^*, running between some of the spots. These differences suggest that in talc the layers nearly always stack one on the other in the same way, whereas in pyrophyllite there are frequent changes in the stacking vector as the stack of sheets is built up. (Brown and Rayner)

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Alteration of micas. Preliminary work on the release of potassium from micas (*Rothamsted Report* for 1964, p. 77) was completed. Five trioctahedral micas, representative of different compositions in the phlogopite–biotite series, and one fine-grained muscovite, were extracted with a solution containing sodium chloride and sodium tetraphenyl boron. The amount of potassium retained in the alteration products was determined at three-weekly intervals, after the micas were washed to remove precipitated potassium tetraphenyl boron. X-ray-diffraction examination of the washed and unwashed product suggests that some potassium is resorbed during dissolution of potassium tetraphenyl boron. After extraction for 4 months, the K_2O content of the trioctahedral micas decreased from 10 to approximately 0·4%; the muscovite, with about the same original potassium content, still contained as much as 2% K_2O.

X-ray-diffraction examination of sodium-saturated, air-dry, oriented samples of the trioctahedral alteration products showed many orders in a rational sequence of basal reflections based on a layer spacing of 12·2 Å. Wetting with water caused expansion, and the materials then gave rational reflections based on 14·8 Å; these spacings compare with a layer spacing of 10 Å in the parent micas and show that when potassium is removed the micas expand to form vermiculites in a manner apparently similar to the natural weathering of soil mica. The altered fine-grained muscovite gave a complex diffraction pattern with several unusual features.

Structural formulae were calculated from the chemical compositions of the altered micas, assuming no change in the oxygen content of the unit cell. The lattice charge arising from isomorphous substitution, calculated from the sum of interlayer ions (Na + K), decreases from about 1·9 per 22 oxygen atoms in the micas to about 1·6 in the altered materials. This change is accompanied by the oxidation of a substantial proportion of the iron when there is much ferrous iron in the lattice. A more precise assessment of the effect of this oxidation on the lattice is being attempted. (Brown and Newman)

The relation between potassium–oxygen bond length and the release of potassium by micas. In their study of the artificial weathering of micas, Raussel-Colom and co-workers showed that micas differ in the ease with which they release potassium (*Experimental Pedology* (1965) ed. E. G. Hallsworth and D. V. Crawford, Butterworths, London). The potassium–oxygen bond length is not the same in different micas, and we have therefore tested the possibility that this bond length is correlated with ease of release of potassium. Raussel-Colom and his collaborators give data for the relative ease with which potassium is released by eight chemically analysed trioctahedral micas. We calculated the b axis dimensions of the micas from their chemical compositions, using Radoslovich's formula (*Amer. Miner.* (1962) **47**, 617); the formulae of Donnay and collaborators (*Acta Crystallogr.* (1964) **17**, 1374) were then used to calculate atomic coordinates and interatomic distances. The calculated potassium–oxygen bond lengths range from 2·881 to 2·983 Å for the shorter and from 3·400 to 3·486 Å for the longer bonds. There is no correlation between these bond lengths and the ease of release of potassium given by Raussel-Colom *et al.*

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If the assumptions inherent in the calculation of atomic co-ordinates are valid, therefore, the potassium–oxygen bond length is not of critical importance in relation to the release of potassium. (Brown and Rayner)

Clinoptilolite. The occurrence of clinoptilolite, a zeolite mineral, in a variant of the Harwell series from Halton in Buckinghamshire, was reported last year (*Rothamsted Report* for 1964, p. 74). It was suggested that this mineral might be responsible for the unusual potassium-releasing properties of a Harwell soil from Bushey Leys, Ellesborough, Buckinghamshire (Arnold & Close, *J. agric. Sci.* (1961) **57**, 295–304). The occurrence is not an isolated one, as clinoptilolite has now been found in the soil studied by Arnold and Close. As in the Halton soil, the clinoptilolite occurs mainly in the fine silt and coarse clay fractions; mica occurs chiefly in the coarse clay, and montmorillonite is dominant in the fine clay fraction. (Brown)

The potassium-supplying power of the different size fractions of the Halton soil is being investigated in collaboration with the Chemistry Department. The distribution of the various minerals between the different size fractions may allow the relative potassium-supplying power of the individual minerals to be estimated. (Weir and Talibudeen, Chemistry Department)

Potassium-supplying power of some soil parent materials. This work was done in the early 1950s to assist in grouping the glacial drifts that form the parent materials of some of the important soil series of the Preston sheet then being mapped by the Soil Survey of England and Wales. Field evidence suggested that the drifts are derived from Triassic and Carboniferous rocks and that they should be divided in three groups—Triassic, Carboniferous and mixed. Because of its agricultural significance, an attempt was made to differentiate the drifts on the basis of their long-term ability to supply potassium, as measured by successive extraction with cold 4*N*-hydrochloric acid. Thus determined, the Trias-derived materials have a larger potassium-supplying power than those derived from the Carboniferous, and, as would be expected, the mixed drifts fall between the other two.

Mineralogical examination showed that the differences in potassium-supplying ability were not related to the potassium-bearing minerals in the sand fraction, but semi-quantitative determination of the clay minerals showed a strong positive correlation between the potassium-supplying power of the clay fractions and the amount of mica in the clay fraction. The study showed that the drifts are derived from two main sources that have been mixed in differing degrees, and broadly confirmed the separations arrived at by field examination. The Triassic drifts, the clay fractions of which are richer in mica, give to soil parent materials to which they contribute a potassium-supplying power superior to that of the Carboniferous-derived materials. (Brown and the late C. J. Tapp, Soil Survey)

Prediction of release of non-exchangeable potassium to crops. In response to a query from the Grassland Research Station for a simple method to

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predict the ability of soils to provide crops with potassium over long periods, we statistically analysed the data obtained by Arnold and Close (*J. agric. Sci.* (1961) **57**, 295–304) on seventeen British soils.

Arnold and Close state that "the main reason for the variations in the K-releasing powers of the soils was traced to differences in the amount and potassium content of the finer clay fractions". The separation of enough fine clay for chemical analysis takes a long time, so we analysed the data to see whether a more readily determined parameter could be used to predict the extent of release of non-exchangeable potassium. The calculations show that the product of the amount and potassium content of the more readily obtainable $<2\ \mu$ esd fraction gives almost as good a prediction of potassium-releasing power ($r = +0{\cdot}794$) as does the product of these two quantities for the $<0{\cdot}3\ \mu$ fraction ($r = +0{\cdot}893$) and the $<0{\cdot}1\ \mu$ fraction ($r = +0{\cdot}874$). The difference between the correlation coefficient for the $<2\ \mu$ fraction and those for the finer fractions are not significant at the 10% level. Preliminary work suggests that better correlation is obtained by including a factor for the cation-exchange capacity of the soil, but the data are too few for this to be adequately tested. It is hoped to extend this work to more soils and to test the relevance of the factor for cation-exchange capacity. (Brown and Dunwoody, Statistics Department)

Calcium phosphates. At the request of M. Webber, Chemistry Department, three samples of synthetic hydroxyapatite were examined for impurities, and to determine the limit of detection of octacalcium phosphate in hydroxyapatite by X-ray powder diffraction. In only one of the three samples were reflections additional to those of hydroxyapatite detected; reflections attributable to calcite ($CaCO_3$) and portlandite ($Ca(OH)_2$) were given by a sample obtained from Scottish Agricultural Industries. Although less than 2% octacalcium phosphate can be detected in hydroxyapatite, as much as 5% could not be detected in a mixture that had been ball-milled; presumably the octacalcium phosphate was destroyed by ball-milling, possibly by conversion to hydroxyapatite. (Brown)

Clay minerals in weathered tuffaceous mudstones of the Old Red Sandstone from the Raglan district of Monmouthshire. The clay minerals of five samples of material from the site of a former landslide in the Raglan district of Monmouthshire, probably derived from tuffaceous mudstones, were examined at the request of H. Humphrey and Son Ltd., consulting engineers for the M6 motorway in South Wales. On the basis of optical examination clay minerals in the tuffaceous mudstones of this area have been classed as kaolinite (Geology of the country around Monmouth & Chepstow; *Mem. Geol. Surv. U.K.* (1960) HMSO, p. 36). We find two of the five clays to be pure montmorillonites, another two dominantly montmorillonite and the fifth to be a mixed-layer mica–montmorillonite with about equal proportions of swelling and non-swelling layers. Chemical analyses are in accord with these identifications. Electron microscopy shows that the montmorillonite consists of large thin plates that are very similar to those of Wyoming bentonite; the chemical composition also is similar to that of montmorillonites from Wyoming bentonites. (Brown)

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Occurrence of jarosite minerals in soils of reclaimed mangrove swamps in Guinea. In connection with J. Coulter's (Chemistry Department) F.A.O. mission to study the reclamation of land for rice growing in the Coastal Belt of Guinea, samples of yellowish powdery segregations from acid sulphate soils ("cat clay") were examined by X-ray powder diffraction. From a site in the Kawass area, which has been free of sea water for many years, the material was jarosite, $KFe_3(SO_4)_2(OH)_6$. Material from a site in Ile de Kabak, which is flooded regularly with sea water, gave a powder pattern similar to that of natrojarosite, $NaFe_3(SO_4)_2(OH)_6$. These identifications were confirmed by chemical analyses. (Brown)

London Clay: Thames mud. Clay minerals in samples of London Clay and "oxidised" Thames mud were identified for Dr. Kitchener, Royal School of Mines, Imperial College of Science and Technology, London. The London clay contained montmorillonite (50%), mica (30%) and kaolin (20%). The X-ray pattern of the clay minerals of the "oxidised" Thames mud was rather weak, partly because of dilution with quartz (40–50%) and calcite (10%). The clay fraction consists of a swelling mineral, probably montmorillonitic, mica and kaolin in the proportions 5 : 3 : 2. (Brown)

Soil Genesis

We completed work on the origin of the Winchester and Patching Series from the Arundel area, Sussex. A mineralogical study of acetic acid-insoluble residues from all zones of the Upper Chalk has allowed us to recognise with greater certainty those parts of the soils that are derived from the Chalk, and contrary to our report of last year, shows that Chalk-derived clay minerals can be recognised in the Winchester Series.

The sand fractions of residues from all zones of the Upper Chalk are composed mainly of flint, opaline silica and collophane, with minor amounts of detrital minerals such as quartz, mica, zircon, tourmaline and sillimanite, and authigenic limonite, glauconite, pyrites, apatite, felspar and barytes. The silt fractions (2–50 μ) are similar in composition, but contain more quartz (often authigenic), mica and authigenic felspar and apatite. The clay fractions contain montmorillonite, mica, quartz and apatite. The morphology of the Chalk montmorillonite is unusual for a clay mineral of sedimentary origin; it is composed of thin laths usually randomly disposed, but often arranged in parallel groups set at angles of 60 degrees to other groups, thus forming pseudocrystals. Apatite occurs as short prismatic euhedra, and is entirely authigenic. In clay fractions of many of the zones the quartz occurs as rounded detrital grains, but in the *testudinarius* zone it often forms delicate skeletal crystals resembling those of ice; in the *pilula* zone at one locality, some of the detrital grains have parallel elongate overgrowths that are crystallographically continuous with each other and with the grain centres.

Many of these minerals were recognised in all horizons of the Winchester and Patching soils, but they are especially common in the extremely clay-rich horizon of the Winchester series that occurs immediately above the

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Chalk. The sand fractions from this horizon contain flint, collophane, sillimanite and limonite; as in the samples from the Upper Chalk, the limonite frequently occurs in cubes and octahedra that are pseudomorphs after authigenic pyrites. The apatite and some of the montmorillonite and quartz in the clay fraction are also morphologically similar to the distinctive varieties present in the Chalk. (Weir and Catt)

Clay Physics

Work on the properties of cohesive soils was continued. The water contents and volumes of soils of about 50% clay content, selected from a wide range of Soil Series, were measured at pF levels of 2, 4 and 6. Hysteresis differences were eliminated by bringing the samples to equilibrium at pF 2, with periodic disturbance by remoulding, and from this unique state the shrinkage with decreasing water content was measured at pF 4 and 6. The shrinkage was compared with other characteristics of the soils, e.g., specific surface areas, Atterberg limits and the contents of calcium carbonate, organic carbon, silt, clay minerals and extractable iron. The work is not yet complete, but thus far it seems that there is no relation between shrinkage and montmorillonite content, although there is a striking positive correlation between shrinkage between pF 4 and 6 and the external specific surface. The external surface depends mainly on the clay content and on the distribution of particle sizes in the clay fraction, so in cohesive soils of different clay contents the extent of shrinkage between pF 4 and 6 should also correlate with surface area. There is some evidence that this is so, but more work is needed.

From field infiltration experiments, Stirk showed that cultivated cohesive soils, which show a tendency to crack in dry weather, absorb water slowly below pF 4·5, but that above this value the infiltration rate increases rapidly (*Aust. J. agric. Res.* (1954) **5**, 279). He concluded that the rapid increase occurred because deep cracks formed below the cultivated surface. The positive correlation between the amount of shrinkage between pF 4 and 6 and the external specific surface area suggests that cohesive soils of large external specific surface area would be particularly subject to cracking in dry summers, with the consequent possibility of soil components being carried down cracks by heavy rain. (Greene-Kelly)

Spectrochemistry

Work on the concentration of trace elements with exchange resins continued, the object being to separate as many trace elements as possible from the principal elements in soils and sediments, i.e., Al, Fe and Si (*Rothamsted Report* for 1964, p. 78). Our early work indicated that recoveries of Mo, Pb, Sn and Zn were quantitative, but further work has shown that the method is not completely reliable. The method is based on retention of the trace elements as chloro-complexes on an anion-exchange resin, which is then ignited with a few milligrammes of Al_2O_3 to act as carrier and to provide a solid concentrate suitable for spectrochemical analysis. Although losses may occur during dry-ashing, the ease with which

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the solid concentrate is obtained by this method makes it especially valuable in spectroscopy. The low recoveries we obtain are due neither to elution of the elements from the resin nor to their volatilisation during ashing; they probably arise from absorption by the material of the crucible in which the resin is ignited; methods of avoiding this are being examined.

Of the elements studied Mo and Pb are least strongly retained by the resin; for these a concentration factor of 50 is possible, but much greater concentration is possible for Ag, Bi, Cd, Sn and Zn, which are more strongly retained in the resin. (Le Riche)

Geochemistry of the Yorkshire Lias. Most of the soils in south and east England are developed on sediments, and a knowledge of the geochemistry of these parent materials is a pre-requisite to a chemical study of the soils. This knowledge involves assessing the conditions of deposition of the sediment—for example, the Eh, pH, depth of water, salinity, palaeoclimate, position of shorelines and the nature of the source rock. Our earlier work on the geochemistry of the Lower Lias in southern England and South Wales (*Rothamsted Report* for 1957, pp. 72–73) was extended to the Liassic sediments exposed on the Yorkshire coast, which merit study because the coastal cliff sections of north-east Yorkshire provide good exposures of almost the whole succession, enabling both vertical and horizontal geochemical variations to be studied and compared with formations of the same age in southern England.

Variations in the conditions of deposition were followed by determining the vertical distribution of trace elements throughout the succession; their association with major constituents—organic matter, carbonate, sulphides and detrital material was also studied. In Yorkshire the Lower Lias was deposited under less anaerobic and generally more acidic conditions than in Dorset, Somerset and Gloucestershire. Only during the deposition of the very organic Jet Rock and Bituminous Shales of the Upper Lias in Yorkshire were conditions sufficiently anaerobic to be comparable with those of the Lower Lias in southern England. Even so, the Jet Rock and Bituminous Shales contain much less Mo than in the south, where the concentration reaches toxic amounts. Co-precipitation with sulphides is an important factor in the concentration of most minor elements in the reduced sediments of the Upper Lias in Yorkshire; Cu is associated with organic matter, and Cr, V, Ti, Zr and Ga are predominantly associated with detrital minerals. (Catt, Gad, and Le Riche)

Soil Chemistry

Partial sterilisation of soil. Work on partial sterilisation (*Rothamsted Report* for 1964, p. 79) was continued. To compare the effects of different methods of partial sterilisation on the mineralisation of soil organic matter, soil samples were treated in three ways—either exposed to pure chloroform vapour, exposed to 2 megarads of gamma radiation (a sterilising dose) or oven dried at 80° C for 24 hours. Immediately after treatment part of each sample was inoculated and incubated, and the evolved CO_2 determined; the remainder of each sample was re-exposed to treatment

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before being inoculated and incubated. About the same amount of organic carbon was mineralised after one or two treatments with chloroform vapour, but with both oven drying and irradiation the second treatment made more of the organic matter decomposable. With a labelled soil (a calcareous soil incubated for 1 year in the field with ^{14}C labelled ryegrass), the organic carbon mineralised by the second treatment was lightly labelled relative to that mineralised after a single treatment. This suggests that in addition to the lethal effect on the heavily labelled biomass, both oven drying and irradiation alter other fractions of the soil organic matter, thus increasing the rate of mineralisation of both biomass and non-biomass C. (Jenkinson)

The accumulation of organic matter in uncultivated soils. The study of the accumulation of organic matter in Broadbalk Wilderness (*Rothamsted Report* for 1964, p. 80) was extended to Geescroft Wilderness. In 1885 a part of Geescroft, which up to then had been arable and used for manurial experiments, was fenced off and allowed to revert to woodland. By 1965 the net gain of nitrogen by the top 27 in. of soil (corrected for changes in the bulk density of the soil) was 1060 lb per acre, only 27% of that gained by the wooded part of Broadbalk Wilderness during almost the same period. (In 1885 the organic contents of Broadbalk and Geescroft were almost the same.) The net gain of organic carbon in the top 27 in. of Geescroft Wilderness was 20,700 lb/acre, compared to gains of 45,400 and 43,900 lb/acre for the wooded and stubbed parts, respectively, of Broadbalk Wilderness. Geescroft and Broadbalk Wilderness are both sited on the same soil series (Batcombe); they are less than half a mile apart, and occur on almost the same level with about the same slope, yet in 80 years one has gained more than twice as much soil organic matter as the other. The leaf fall in 1965 was the same in both sites, equivalent to 1,300 lb organic carbon per acre, so that differences in the annual addition of organic matter to the two soils cannot explain the contrast between the accumulation of organic matter in the two sites. Phosphorus deficiency cannot have limited the accumulation of organic matter in Geescroft Wilderness because there is no significant difference between the amount of organic matter in soil from the part treated with superphosphate during the period 1847–78, which contains 780 ppm phosphorus in the top 9 in., and the organic content of soil from the part that has never been manured, which contains 560 ppm phosphorous.

The difference in pH between the Geescroft and Broadbalk sites seems to be the only factor that could account for the different rates of accumulation of organic matter. Broadbalk was calcareous in 1881, and the Wilderness still contains calcium carbonate. In 1883 the pH of the top 9 in. of Geescroft was 7·1; by 1904 this had fallen to 6·1 and by 1965 to 4·5. Soil organic matter is probably more stable in calcareous Broadbalk than in non-calcareous Geescroft, and so accumulates faster there. (Jenkinson)

The determination of organic phosphorus in soil. As part of our investigation of the accumulation of organic matter in uncultivated soils we planned to follow the accompanying changes in organic phosphorus, and four

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methods of measuring soil organic phosphorus were compared. Table 1 gives the results. Method A, in which organic phosphorus is taken as the difference between the phosphorus soluble in cold $2N$-H_2SO_4 before and after ignition, always gave the largest value. In methods B and C, organic phosphorus is taken as the difference between the phosphorus soluble in

TABLE 1

Organic phosphorus in soils from Broadbalk Wilderness, as measured by 4 different methods

Soil	Organic Carbon, %	Organic phosphorus, ppm Method A	Method B	Method C	Method D
0–9 in. sampled in 1881	1·06	250	180	180	150
18–27 in. sampled in 1881	0·46	200	70	80	40
0–9 in. sampled in 1964	2·89	420	270	290	200
18–27 in. sampled in 1964	0·55	290	90	140	10

Method A Williams, C. H., Williams, E. G. & Scott, N. M. (1960), *J. Soil Sci.* **11**, 334–346.
Method B Legg, J. O. & Black, C. A. (1955), *Proc. Soil Sci. Soc. Am.* **19**, 139–143.
Method C As B, but ignition temperature 550° instead of 240° C.
Method D Mehta, N. C., Legg, J. O., Goring, C. A. I. & Black, C. A. (1954), *Proc. Soil Sci. Soc. Am.* **18**, 443–449.

hot concentrated HCl before and after ignition. In both B and C slightly more phosphorus is dissolved after ignition than in method A, but as much more inorganic phosphorus is dissolved from the unignited soil, methods B and C give considerably smaller values for organic phosphorus than method A. The differences between the amounts of phosphorus extracted by hot HCl and cold $2N$-H_2SO_4 from the unignited soils are 130 and 220 ppm for the 1964 topsoil and subsoil respectively; as the subsoil contains much less organic matter, these differences are unlikely to represent organic phosphorus hydrolysed by hot HCl but not by cold dilute H_2SO_4. A more plausible explanation of the discrepancy between methods A and B (or C) is that a fraction of originally insoluble inorganic phosphorus is rendered soluble in cold $2N$-H_2SO_4 by ignition, whereas this fraction is soluble in hot concentrated HCl whether or not the soil has been ignited; with method A this fraction would be reported as organic phosphorus. In method D the soil is extracted with hot concentrated HCl, followed by cold and hot NaOH; the difference between the total and inorganic phosphorus in the combined extracts is called organic phosphorus. Values obtained by this method were lower and more variable than with the other three methods. The total amount of phosphorus extracted did not differ greatly from that extracted from ignited soil by concentrated HCl (methods B and C), suggesting that extraction was complete, and that this method gives low values for organic phosphorus because organic phosphorus is hydrolysed during extraction. The discordant results in Table 1 show that caution is required in interpreting the results from currently accepted methods of determining organic phosphorus. (Jenkinson)

Mechanism of podzolisation. No standard procedure for collecting and preserving leaf material has been adopted by the various investigators of

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the iron-mobilising properties of aqueous leaf extracts. We therefore determined the extent to which the properties of the extracts change during senescence, and how they are influenced by the method of drying. The amounts of ferric oxide dissolved by extracts of autumn-fallen oak, beech and larch leaves were compared with the amounts dissolved by extracts of leaves collected 2–3 weeks before leaf fall. Similar measurements were made on extracts prepared from freshly gathered leaves before and after freeze-drying and oven-drying at 105° C. In general, the capacities of extracts of autumn-fallen leaves to dissolve ferric oxide were not significantly affected by either method of drying. Apart from this there was little uniformity in the results. Freeze-drying had almost no effect on picked beech leaves, whereas oven-drying increased the amount of iron dissolved five-fold. Oven-drying also increased the activity of picked larch needles, but less than with beech leaves. With larch and beech there were no significant differences between the activities of undried picked and fallen leaves, but fallen oak leaves were more active than those picked some weeks earlier. This difference was reflected in the results of the ageing experiment, in which undried fallen leaves were maintained at constant moisture content for 28 weeks before preparing the extracts. (No mould growth was observed during this experiment.) The activity of the oak leaves increased to nearly three times its original value during the first week's ageing and decreased to about half this maximum in the next 3 weeks. Thereafter the activity decreased slowly, but after 28 weeks it was still slightly greater than the original value. The activity of the beech leaves increased slightly in the first week, fell to about the original value after 4 weeks and then remained more or less constant. In the first week the activity of the larch needles decreased to less than half the original value, and continued to decrease slowly for the rest of the period.

There was no consistent relationship between the polyphenols content of the water extracts, as determined either with Folin–Denis reagent or by visual assessment of paper chromatograms, and their capacities to dissolve ferric oxide. Previous work showed that the contribution of polyphenols to the iron-dissolving capacities of leaf extracts may vary directly or inversely with their degree of condensation. The extent to which dissolved iron compounds and organic matter are sorbed on ferric oxide, with consequent inhibition of solution of the latter, is similarly influenced by the molecular complexity of the polyphenols; the continuous variation of all these factors is no doubt responsible for the absence of a single trend in the results of these experiments. The method of preparation and the state of the leaves when collected must clearly be considered in future work. (Bloomfield, King and Pruden)

Leucoanthocyanins of larch needles. Arising from our work on the mobilisation of iron by leaf extracts, we found that picked and fallen larch needles contain a new leucodelphinidin, a leucocyanidin and the leuco-form of an unknown anthocyanin. A water-soluble leucodelphinidin is coprecipitated with an insoluble calcium compound when an aqueous extract of larch leaves is allowed to stand for a week or so. The leucodelphinidin seems to be a glycoside in which the sugar groups mask the

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phenolic function. Together the leucodelphinidin and the calcium compound constitute 2–3% of the dry weight of the larch leaves, corresponding to 10–15% of the water-extractable matter. (King)

Numerical Classification

The numerical method of classification used for 23 Glamorganshire soils (*Rothamsted Report* for 1962, p. 76) gives a family tree of the soils. A better representation of their relationships is obtained from the latent roots of their similarity matrix. The similarity matrix is adjusted to make the sums of its rows and columns zero; the first two latent vectors then give a map of the relationships between the soils. A representation in more than two dimensions is obtained by taking further vectors. A computer programme was written to use the similarity matrices prepared by the Orion classification programme CLASP for calculating the vectors for a representation in as many dimensions as required.

A calculation of this kind on the 91 horizons distinguished by the surveyor in the 23 Glamorganshire soils showed that the most important vector determining the positions of the points representing the horizons was very similar to the first vector found for the soils as a whole. These vectors separate the group of acid brown earths from the rest. The second most important vector in the representation of the horizons depends on their sequence in the profile. These two factors, sequence and acidity, are confused in a family tree of the horizons but are nearly completely separated from one another in this representation. This separation may be compared with the practice of surveyors of first distinguishing horizons, according to their sequence, as major kinds A, B and C, and then on the basis of features that occur only in some soils, e.g. gleying. (Rayner with Gower, Statistics Department)

Apparatus and Techniques

Spurious effects may be observed in X-ray diffractometry, even when pulse-height selection is used with a proportional counter. Some of these effects were described by Parrish (*Advances in X-ray Analysis* (1965) **8**, pp. 118–133. New York: Plenum Press). An effect not reported by Parrish was observed during the examination of vermiculites produced by removing potassium from micas. This occurs when specimens that give very strong first order reflections in the region 5–10° 2 θ, such as a well-oriented aggregate of mica or vermiculite, are examined at high sensitivity of the recording circuits with the pulse-height analyser set to pass about 90% of the characteristic radiation. For example, a weak reflection was observed at a $2\theta \simeq 4°$ when a 14·8 Å vermiculite specimen was examined with iron-filtered radiation from a cobalt target. This would be interpreted as a reflection with $d = 25$ Å were it caused by the characteristic CoKα radiation. The peak seems to be caused by white radiation, and to arise because sufficient pulses from the white radiation have amplitudes that allow them to pass the pulse-height-analyser window when it is set for the characteristic radiation.

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A stabilising circuit that controls the tube current to $\pm 2\%$ was fitted to the Raymax 60 generator used for diffractometry. A reference current derived from the X-ray tube current is used to monitor the X-ray output; this is sufficiently precise to replace the previously used more elaborate monitoring system, which measured the diffracted intensity from an aluminium foil with a counting tube. (Brown and Farrow)

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