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Report for 1958

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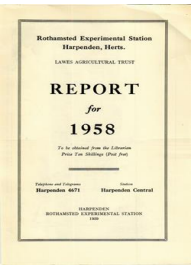
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Pedology Department

A. Muir

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PEDOLOGY DEPARTMENT

A. Muir

Mineralogy

Iddingsite

The study of this mineral has been completed (3.3). The only major change in the interpretation of the X-ray photographs as given in the report for 1956 is that reflexions then supposed to arise from a super-lattice have now been shown to be more probably due to goethite in two subsidiary orientations in addition to the main orientation parallel to the original olivine. (Brown and Stephen.)

Jarosite

The claim to the first record of this mineral in soils has proved an error, because it had earlier been reported by E. M. Chenery (*The Soils of Central Trinidad*, 1952). However, the study of the mineral has continued because the data in the literature on cell constants of the end members (the K and Na varieties) conflict. An examination of analysed samples of jarosite (K-member) and natrojarosite kindly supplied by Dr. A. W. G. Kingsbury, Oxford University Museum, has indicated a relationship between the cell constants of the mineral and its relative potassium and sodium contents. Assuming this to be linear for the *c* crystallographic dimensions, the jarosite-type mineral from Bow Brickhill should contain the alkalies in the proportions 72 per cent K and 28 per cent Na. Chemical analysis gives 74 per cent K and 26 per cent Na, which suggests that the alkali cation content of the members of the series jarosite–natrojarosite could be quickly and accurately determined from measurement of the *c* dimension by X-ray diffraction. (Brown.)

Loess-like deposits in southern England

Mineralogical and other studies at Rothamsted and elsewhere have shown the probability that many soils are developed, at least in part, in loess-like material. In the Chilterns the rich assemblage of heavy minerals found in the silty surface horizons of soils on plateau drift contains species which are rare or absent from the substrata. The origin of the added minerals cannot yet be deduced, but glacial and glacio-fluvial drift are obviously possible sources. A mineralogical comparison of Chalky Boulder Clays from Hertfordshire and Buckinghamshire, on either side of the Chiltern Hills, showed a general predominance of garnet, epidote and hornblende over tourmaline and staurolite, which is similar to assemblages previously reported for glacial drifts of similar age in East Anglia. Such deposits or their outwash could have supplied all the minerals characteristic of the Chiltern silty deposits, except possibly for a chlorite which is never important in the glacial drifts but is prominent in the silts. The type of chlorite is, however, identical in both

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materials, and it may be locally more abundant in the boulder clays. The department is also co-operating with the Soil Survey in the examination of a similar problem in Somerset (*Soil Survey of Great Britain, Reports 9 and 10*). (Stephen.)

Micropedology

It is becoming more and more apparent that a straightforward soil profile description is not a sound basis for soil classification. The techniques of thin-section investigation have brought out features that had hitherto escaped notice or were only vaguely suspected, but are a distinct aid to classification.

The study of thin sections of the Chiltern soils referred to previously (*Rep. Rothamst. exp. Sta. for 1956*) has been completed. With certain soils developed in loess-like material, there is good evidence for clay migration down the profile, which in part accounts for the considerable change in texture in the subsoil. An examination of several soils developed in silt material overlying Carboniferous Limestone on the Mendips showed that they, too, possess micromorphological features indicating clay migration and deposition with the formation of textural-B horizons. Such features suggest that the soils are related to grey-brown podzolic soils (Frei and Cline (1949), *Soil Sci.* **68**, 333) or parabraunerde (Kubiena (1956), *Eiszeitalter und Gegenwart*, **7**, 102). A preliminary study of some soils of the group " red and brown calcareous soils " overlying Oolitic Limestone on the Cotswolds indicated the possibility of their assignment to such groups as brown rendzinas, ferritic braunerde and terra fusca (Kubiena (1953), *Soils of Europe*). Dr. R. R. Storrier showed that the soils on the Banbury ironstone were of the ferritic braunerde type, whereas others in that area could be considered as braunerde and rendzinas.

Thin sections of some "red soils" from various parts of Africa were prepared for comparison. In particular, it was hoped that a study of the red earths from Tanganyika might help to explain why they set so very hard when dry and are so abrasive when worked (cf. *Rep. Rothamst. exp. Sta. for 1950*, p. 46). These soils contain much coarse sand and clay and little fine sand and silt. Thin sections of clods from the top foot show the coarse sand to be dominantly angular quartz. There is evidence of clay movement and its accumulation on the walls of pore spaces. It appears that intense rainfall in the wet season promotes clay shift that is confined mainly to the upper layers of the soil and results in most of the pores in the subsurface soil becoming clogged with close-packed clay. The clay is mainly non-swelling kaolin, so that a massive structure is produced, and it is suggested that this structure of dense, massive peds studded with firmly held angular quartz grains would be highly abrasive. (Stephen and Osmond.)

Clay Mineralogy

The samples of soils examined during the year for their clay mineral content have come from various countries. British samples included soils and parent materials from Somerset, parent materials

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from Shropshire, and clays and limestones from various parts of England.

The parent materials from Somerset range from the Devonian to the Lower Jurassic, and generally showed moderate or dominant mica. The presence of an appreciable amount of sepiolite in a Triassic marl sample was noteworthy. Parent materials from Shropshire included samples from Upper Coal Measures, Downtonian and Dittonian. A remarkable variation in the clay composition of the Downtonian was indicated by the presence of a considerable amount of montmorillonite in all the Ledbury Marls examined and its complete absence in the other Downtonian samples which consist in the main of mica and kaolin. (Du Feu.)

It has been suggested that the mineral palygorskite, which has been reported as widespread in Middle East soils (*Rep. Rothamst. exp. Sta. for 1957*, p. 67) might be of pedogenetic origin. By courtesy of Mr. R. H. S. Robertson it has now been possible to examine some Iranian rock samples and a clay undoubtedly derived from igneous rock material. Some of the sedimentary rocks showed the presence of palygorskite, but there was no sign of it in clay from igneous rocks. This suggests that the palygorskite in the soils is derived from their parent rocks and is not formed by contemporary weathering or pedogenetic processes.

The Ukiriguru soil catena in Tanganyika is being studied. This sequence of soils derived from granite ranges from shallow red brown soils just below a tor, through rather sandy soils, some of which develop an extremely hard pan on drying, down to the heavy black mbuga clays. The soils on the upper slopes contain largely kaolin and mica; in the sandy soils montmorillonite comes in at depth, and together with slight sodium saturation undoubtedly contributes to the pan formation; the montmorillonite increases in quantity as the mbuga soil is approached, and in this is by far the dominant clay. (Du Feu and Muir.)

Effect of sodium hydroxide attack on montmorillonites

Previous work on the Black Jack Mine beidellite specimen suggested that the small-particle-size material seen in electron micrographs and the 350° C. endothermic d.t.a. peak might be from an aluminous impurity. To test this hypothesis samples of the beidellite and three pure montmorillonites, Wyoming, Redhill and Unter-Rupsroth, were extracted for 4 hours at 100° C. in 0·5*N*-sodium hydroxide. The beidellite, of which about 20 per cent dissolved, was the only one appreciably attacked. The others showed some selective extraction of silica, but the amounts were too small to make a significant difference in the total analysis figures. Calculation of the half-unit cell contents from the analysis of the extracted beidellite showed a slight shift towards an ideally dioctahedral mineral and reduction in inter-layer charge. The change was small, however, and the dissolved material could not be called an aluminous impurity. It was difficult to say whether it was the small-particle-size material that had been dissolved. Where the sample dispersed easily this appeared to be so, but where the material did not disperse, electron micrographs showed considerable attack on large flakes. Extraction of the beidellite showed no change in the 350° C.

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endothermic d.t.a. peak, and further work showed that such a peak is a common feature in minerals of this type. There seems no reason to doubt that the beidellite is a pure end-member of the montmorillonite–beidellite series. The work has also confirmed the use of sodium hydroxide extraction for the removal of fine-grained silica and to some extent alumina impurities for the calculation of unit cell contents, but it is very doubtful whether it can be regarded as a useful-pre-treatment for samples. (Weir.)

Electron microscopy

The technique of spraying on the grid has been used successfully with some montmorillonite minerals. It can be used as an exploratory method complementary to optical microscopy, and with a range of magnification of 200–20,000 the whole sample can be surveyed initially and then areas selected and examined with increased magnification. The Unter-Rupsroth montmorillonite proves to have an undisturbed structure similar to that of the beidellite, but the electron micrographs are less spectacular, because the flakes are equi-dimensional rather than lath-shaped and do not dry into such clear-cut fans.

Considerable work has been done on the electron microscopy of unshadowed montmorillonite mounts. At first a magnification of 5,000 was thought best for these very thin materials and lack of contrast was considered to prevent successful results at 20,000 or 40,000, but this has proved mistaken. The use of very thin carbon films has improved contrast, but the physical state of the sample is the most important factor. Washing and high-speed centrifuging removes what appears to be a decomposition product of the clay minerals, which otherwise blankets the entire mount and drastically reduces contrast. Further work will be needed to define the chemical composition of this material, the conditions under which it is produced and its volume relations to the original sample. The presence of such material has probably been overlooked previously in shadowed mounts because sufficient relief remained for shadows to be cast, and the contrast between metal and shadow was the dominant feature. In addition, the tendency of flakes and flake-shadows to disappear into the mount has frequently been attributed to depression of the collodion when drying. Successful unshadowed pictures of the Camp Berthaux montmorillonite have recently been taken and show the irregular shapes and re-entrant angles mentioned by Méring (*Bull. Soc. franc. Miner. Crist.* **79** (1956), 515). (Nixon and Weir.)

Physical Chemistry of Soil Minerals

Interaction of water and clays

The energy distribution in such systems as kaolinite and water, where swelling is small, is being investigated to try to establish how adsorbed layers of water are related to bulk water. Evidence is accumulating that the film thickness at which the liquid-vapour interface can be said to resemble that obtained with bulk water is greater than has been previously supposed.

It has been shown that the suction of a non-swelling system is a

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differential function of the liquid–vapour interfacial area, and hence the heat-of-immersion method can be used to measure pF. This method has now been successfully used down to a pF of 4, but at this stage there were difficulties because a variable blank heat breaks the thin glass containers. R. C. Gallavan has investigated the cause and remedy of this variability and found that a fixed composition of dissolved gases in the water used for immersion is necessary to maintain a constant blank heat of breaking. This combined with a new method for breaking the bulbs, and corrections for the degree of filling and the vapour pressure within the bulbs enables the blank heat to be predicted to $\pm 0{\cdot}01$ joule. To make use of this accuracy the calorimeter sensitivity has been increased four times, so that the temperature sensitivity is now 5 micro degrees per mm. deflection. Some of the less-satisfactory work is now being repeated, with a possible extension of the suction range measurable in conjunction with discontinuities in the build-up of thick water films on clays (*Rep. Rothamst. exp. Sta. for 1957*, p. 69).

Studies were continued on swelling clays such as montmorillonite, with which an appreciable amount of energy is associated with swelling and must be allowed for. The heat of immersion of montmorillonite samples plotted as a function of water content show inflexions directly related to the points where the interlayer water thickness changes. From these changes it is hoped to obtain data on the energy of swelling. (Greene-Kelly and Gallavan.)

Optical properties of clays

During the recent visit of R. Greene-Kelly on a Royal Society Exchange Fellowship to the Academy of Sciences, U.S.S.R., Prof. Deryaguin suggested that it would be of interest to study the optical properties of montmorillonite flakes in salt solutions and to deduce the optical properties of the interlayer water as a function of film thickness. The results obtained showed that the interlayer water when about 50 Å. thick, had a weak positive birefringence (i.e. of the same order as ice). At Rothamsted Greene-Kelly has continued his study of the optical properties of clays in two main directions with encouraging results: first, the measurement of the birefringence of organic complexes of montmorillonite previously studied by X-ray methods (*Rep. Rothamst. exp. Sta. for 1953*); secondly, an optical examination of different types of expanding and partly expanding clay minerals. A complex of montmorillonite and an organic compound has a birefringence which depends, among other factors, on the orientation of the molecule in the interlayer space. Thus, an isotropic complex can be prepared from a strongly negative silicate mineral and an equally strongly positive interlayer substance. This makes possible the identification of montmorillonite-like interlayer spaces from the optical properties of samples in different solvents, irrespective of the degree of disorder of the mineral. In this way the optical method can be considered a valuable supplement to the X-ray method, particularly when dealing with disordered minerals such as occur in soils. Various disordered mica-like minerals have already been examined with success. (Greene-Kelly.)

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High-pressure synthesis of clay minerals

The setting up of the equipment has proved troublesome, but samples of clay minerals have been successfully synthesized. Specimens of the synthetic clay have been examined by X-rays, D.T.A. and electron microscopy and chemical analysis. The low angle X-ray-diffraction camera (*Rep. Rothamst. exp. Sta. for 1957*) was used to measure the degree of crystallinity of the products. Though there is much less central scatter from the synthetic material than from untreated gels, there is much more than from a well-crystallized natural kaolin. (Rayner.)

Spectrochemistry

Levels of trace elements present in certain English sedimentary deposits, in particular the Gault and Oxford clays and the Lower Greensand, are being studied with the object of relating them to the levels found in soils.

Earlier work on the Lower Lias shales has shown that abnormally high contents of Cu, Mo, Ni and V are related to the amount of bituminous material present. Samples of Oxford clay analysed have again shown that clays with high bitumen content contain larger amounts of these elements, but the enrichment is not as great as in the Lias. Whereas Mo levels of 200 p.p.m. were found in the Lias, the highest amount found in the Oxford clay was only about 20 p.p.m. The levels of Cr and V are in general higher in the Oxford clay than in the Lias or the Gault. The organic material deposited in both the Lias and the Oxford clay was probably potentially capable of accumulating Cu, Mo, Ni and V from the sea-water, and the differences between their present contents arise at least in part from different abundances in the water. The Gault clays examined were much less bituminous, and no evidence was found of enrichment of these elements in them. Apart from these differences, Co, Cr, Cu, Ga, Mn, Mo, Ni and V remained at fairly uniform levels in all three formations. No great variations in trace-element assemblages are likely to occur between similar soils derived from such rocks.

In the Lower Greensand the levels are in general much lower. Specimens ranging from consolidated sandstone to pure silver sand showed large differences in trace-element contents. The former, which contains far more iron oxide, is also much higher in trace elements, although the amounts are still small. It is therefore to be expected that soils on such materials will not only be low in trace elements but that their content will vary more than in soils derived from the clay sediments.

In this connection it is noteworthy that soils from Rothamsted gave consistently higher figures for Ba, Co, Cr, Cu, Ga, Mn, Ni and V than the Woburn soils. (Le Riche and Burnett.)

Soil Chemistry

Effect of soil waterlogging

Anaerobic incubation of a waterlogged soil to which dried grass was added caused part of the iron contained in the soil to become

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extractable by a solution of ammonium acetate (*Rep. Rothamst. exp. Sta. for 1957*). The amount of iron thus mobilized increases with increasing organic matter up to about 20 per cent by weight of the soil. Increasing the organic matter to 30 per cent has relatively little effect on the amount of iron mobilized, although the maximum amount of iron extracted by ammonium acetate does not exceed about half the " free iron oxide " that may be removed from the soil by dithionate or similar treatment.

The solution of the oxides of some minor elements by decomposing plant remains was studied. Cu, Zn and Mn are very readily dissolved, Co and Ni less readily. Molybdenum, as molybdic oxide or as calcium molybdate, is virtually inactive; however, when ferric oxide prepared by precipitation in the presence of molybdate was used as the source of molybdenum, appreciable quantities were mobilized during incubation with dried grass. A similarly prepared co-precipitated material was used in testing the behaviour of vanadium, because vanadium pentoxide is appreciably soluble in water; here again, vanadium was mobilized. The effect with molybdenum may provide a clue as to the way in which this element becomes available in teart soils.

With all the elements studied, the mobilized material passed through cellophane on dialysis, so that the process involves true solution, and is not one of peptization.

Aeration of fermentation solutions of Cu, Zn, Mn, Co, Ni precipitates little or none of the particular element, but when fermented in the presence of ferric oxide, some of the trace element is carried down on aeration with the precipitated ferric oxide. Further, the minor elements are effectively sorbed from the fermentation solutions by ferric oxide. These observations are no doubt relevant to the fact that ferric oxide concretions, or laterite, commonly contain much more trace elements than the surrounding non-concretionary material.

Gleying decreases the total content of minor elements in a soil, but, for most of the trace elements at least, it increases the ratio of the readily soluble to the total amount of the particular element present. In agreement with this, whereas a minor element that has been sorbed on ferric oxide may not be removed by water, the greater part of the sorbed metal may be removed by treatment with a solution of neutral ammonium acetate. (Bloomfield, Greet and Siew Kee.)