

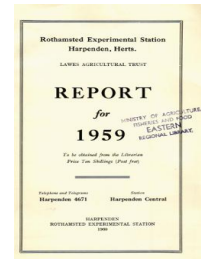
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PEDOLOGY DEPARTMENT

A. MUIR

Dr. Ya. Rusko, an exchange student from the U.S.S.R. (Kiev), spent nine months between the Pedology and Plant Pathology Departments. Dr. S. P. Mitra (Allahabad) has now gone to the United States. Instruction in X-ray techniques was given to Mr. J. W. Vail (Tanganyika) and Dr. A. K. Helmy (Egypt), and Dr. J. S. P. Yadav (Dehra Dun) spent some time studying spectrography. Mr. C. B. Wells (C.S.I.R.O.) will work with C. Bloomfield on carbonate movement in soils.

MINERALOGY

Expanding lattice minerals

(1) In the course of characterising the weathering products of picrite from Shropshire (*Rep. Rothamst. exp. Sta.* for 1957, pp. 65–66), two unusual types of expanding-lattice minerals were studied. A variety pseudomorphous after and derived by weathering from fibrous chlorite occurs in fairly large amounts in the (B)/C horizon of a brown earth at Woodgate, $\frac{1}{2}$ mile south of Corndon Hill. The fibrous character and swelling properties of minerals classified as saponite, bowlingite and diabantite by other workers were demonstrated, and fibre photographs of bowlingite (Cathkin Hills) taken here show a general resemblance to those of the Woodgate soil mineral, but with differences in detail. A lamellar mineral occurring at the base of the soil seems very similar to the layer-lattice silicate found to be a constituent of iddingsite (Brown and Stephen, *Amer. Min.* 1959, **44**, 251–9), giving a disordered stacking of layers, the only regularity, in addition to the parallel and equidistant stacking of the silicate layers, being the parallel alignment of the hexagonal *a*-axes of the half-layers of the mineral. A distinction from the iddingsite material is that the soil mineral expands with glycerol. Various crystallographic considerations suggest that both the fibrous and the lamellar minerals owe their crystallographic order to the inheritance of undisplaced structural units from their parent minerals. The study of such pseudomorphs should give structural information about clay minerals that could not otherwise be easily obtained (see abstract 3.4). (Brown and Stephen.)

(2) An expanding layer-lattice mineral from Baluchistan, Pakistan, was identified as an alleverdite. Chemical analysis confirmed that it was a sodium aluminium silicate and contained only traces of other metal atoms. X-ray-diffraction patterns were identical with those of the type mineral from Alleverd, indicating a mineral with a structure composed of silicate sheets with a thickness of 19.4 Å., separated by layers of intercalated water or organic molecules. The structure was described by Brindley (*Amer. Min.* 1956, **41**, 91). Both the Baluchistan and type alleverdites were formed as skin-like aggregates of flakes, and specimens of natural minerals gave hkl reflections on X-ray-diffraction photographs.

The appearance of these reflexions depended on the previous dispersion state of the specimen, and discrete reflexions were replaced by diffraction bands in patterns from specimens made from suspensions of fully dispersed flakes.

Measurements of the effect of charge density on lattice expansion of alleverdite, beidellite and vermiculite showed that the volume occupied by inter-layer water or organic molecules decreased as the charge density of the expanding layers increased. With inter-layer ethylene glycol the contraction of the lattice was accompanied by reorientation of the glycol molecules. The beidellite had a charge density of $0.46/\text{Si}_4\text{O}_{10}$, intermediate between those of the other two, and showed a complex arrangement of ethylene glycol.

The electron microscope showed that the type mineral was composed of large flakes with folded edges and narrow folded ribbons. The electron micrographs showed that the ribbons were flattened helical spirals, and thus had been present as helices in suspension. B-alleverdite was composed of flakes of equally large area, $10\text{--}40\ \mu$ were characteristic dimensions, but had no ribbons, and the flakes were not folded.

The thickness of the thinnest flakes of alleverdite and beidellite, freed from finely dispersed material, were measured directly from shadowed electron micrographs. The thinnest flakes of alleverdite, calculated from shadow lengths, were $18\ \text{\AA}$. thick, and those of beidellite, $14\ \text{\AA}$., which is direct evidence that flakes consisting of double 2:1 units for alleverdite and single 2:1 units for beidellite were present in suspension. Other experiments demonstrated that alleverdite flakes were easily dispersed completely, but with beidellite, in common with other smectites, this was rarely achieved. Increase in the thickness of the metal shadowing in the micrographs increased the apparent flake thickness, and this in part explained the discrepancy between the thickness of beidellite flakes measured from micrographs, $14\ \text{\AA}$., and from X-ray-diffraction patterns, $9.7\ \text{\AA}$. The existence of the double-layer structure in alleverdite was demonstrated directly in one electron micrograph where the shadows of two $9\text{-}\text{\AA}$. edges merged at either end to form single shadows of an $18\text{-}\text{\AA}$. edge. Double edges were common in the folded alleverdite, and resulted from cleavage along the mica-like interface relieving the stresses developed during the flattening of the helical ribbons. (Weir.)

CLAY MINERALOGY

Several visitors were given instructional courses in X-ray diffraction and its application to clay-mineral studies. To lessen the work involved in future, a series of instruction sheets is being prepared. Eventually it will cover the basic concepts and applications of X-ray diffraction to clay mineralogy, and it will both save time now and provide a permanent record of the course. (Brown.)

G. Brown is editing the new edition of *X-ray Identification and Crystal Structures of Clay Minerals* published by the Mineralogical Society and is contributing the chapter on "Other Minerals".

Soils and sediments

Many clay fractions from soils and sedimentary rocks were analysed. They include samples from Tanganyika (10), Kenya (2), Borneo (15), Basutoland (7), Sudan (6), Yugoslavia (7), Uganda (4), Malaya (4), Swaziland (19) and Britain (84). Of these, 38 were examined under the Colonial Office scheme for analytical assistance to overseas territories. Clay fractions from soils and clay minerals being studied by members of Rothamsted staff and others were also examined.

At the suggestion of P. H. T. Beckett (Oxford) some samples of Cretaceous rocks laid down under desert conditions were examined for palygorskite, but none was found.

After the interesting results on the Downtonian from the West Midlands reported last year (*Rep. Rothamst. exp. Sta.* for 1958, p. 63) many specimens of Old Red sediments were collected from that area. The variability of the clay mineralogy, both laterally and vertically, is being studied in the hope of relating it to sedimentation conditions. The study will also provide useful information on the parent materials of a group of very fertile soils. The specimens so far examined show that the clays are fairly well crystallised. (Du Feu.)

Further investigation of the Ukiriguru catena (*Rep. Rothamst. exp. Sta.* for 1958, p. 63) showed that all the soils of the sequence had granite as their ultimate parent rock. The soil clays in the upper part of the catena are relatively rich in mica, which does not occur in a specimen of weathered granite from the tor. Kaolin is the only clay mineral identified in the rock material. The leaching conditions on the tor are probably such that potash set free from feldspars is immediately removed, thus preventing the formation of mica, which can, however, accumulate in the soils below. (Muir, Du Feu and Stephen.)

Apparatus

Preliminary work showed that the X-ray diffractometer can be adapted for use as a spectrometer for X-ray fluorescence analysis by altering the collimation system. In view of the undoubted importance of this method of analysis, advantage was taken of the adaptability of the instrument to gain some experience of the method.

An attempt was made to build a suitable camera for determining the iron content of materials by non-dispersive X-ray fluorescent analysis. The apparatus would be simpler and less expensive than that normally used. The fluorescent radiation was measured by photometering the X-ray film exposed in the beam. Unwanted scattered radiation interferes seriously, but by using suitable filters and Geiger counters it should be possible to surmount the difficulties.

In modifying the old electron microscope for pedological studies, the first requirement was to obtain an electron beam as narrow as possible. Moderate success was achieved in converting the machine into a two-lens X-ray projection microscope, and a fairly good definition obtained. The present electron beam is in the region of $10\ \mu$, and further work will be done to bring this to $1\ \mu$ or less,

ocused as far outside the lower lens as possible. This will then enable the X-ray microscope to be used as an electron probe.

PETROGRAPHY AND MICROPEDOLOGY

Latosolic red soils and an associated yellow soil from Mount Zomba, Nyasaland, are derived mainly from syenite. The soils, however, have somewhat different mineralogical compositions and, in particular, the yellow soil contains more unweathered primary minerals, e.g., feldspars and hornblende, than the red. This suggests that the soils differ in their weathering regime, or that the red have been weathering for longer. The former possibility seems the more likely, as the soils appear to have a catenary relationship with the red soils occupying the higher topographic sites and the yellow soils occurring in depressions. The humus forms also indicate a difference in weathering regime, mull humus being associated with the red soils, whereas the yellow has a mull-like moder humus cover. Thin-section studies show the breakdown of primary minerals to clay-sized materials, but give no evidence of any translocation of clay within the profiles. This contrasts with red-soil formations from Kongwa and Nachingwea in Tanganyika, derived from material of Basement Complex origin, in which there is evidence of clay migration and its accumulations on the walls of pore spaces (cf. *Rep. Rothamst. exp. Sta.* for 1958, p. 62). The red soils from Zomba appear to correspond closely to the (lateritic) roterdes as defined by Kubiena (*Soils of Europe*, 1953). (Stephen and Osmond.)

PHYSICAL CHEMISTRY OF SOIL MINERALS

Optical properties of clays

The work described last year was continued. In the study of methods to determine the birefringence of clay aggregates, an improved microtome technique enabled uniform clay sections of known thickness to be cut. The birefringence of sections can thus be estimated directly from their optical retardation. With simplification the method is suitable for routine use. Optical studies of the highly disordered hydrous micas showed that these minerals form mixed layer complexes with aromatic molecules and up to half the layers expand. Evidence is accumulating that these expanding layers resemble those of montmorillonites more than those of vermiculites; i.e., if the expanding part of the hydrous mica crystals were regular, they would give X-ray spacings of 18 Å. rather than 14 Å. with glycerol.

Surface studies on clays

Work on several topics mentioned in previous reports was completed, and the results are being prepared for publication.

The increasing use of the B.E.T. apparatus to measure surface areas made it desirable to modify the somewhat tedious procedure. A simple and compact apparatus was designed and built and is proving most satisfactory. Forty to sixty samples can now be measured using one container of liquid nitrogen over a period of a

week, and its accuracy is not significantly less than the more complex unit. The apparatus is being used to study the finest inorganic fractions of soils. (Greene-Kelly.)

Clay mineral synthesis

The apparatus for high-pressure synthesis was much more reliable this year and a series of experiments at 250° and 350° C. yielded products in which crystalline phases could be identified by X-ray analysis, including kaolinite, montmorillonite, analcite, paragonite, boehmite, chlorite and amesite. The products were also examined by differential thermal analysis and by electron microscopy and one by electron diffraction—a method it is hoped to use more often in the future. The products and any particle size fraction thought likely to be a nearly pure mineral, such as the coarse and fine fraction of one preparation which were almost pure paragonite and analcite respectively, were analysed chemically.

The paragonite formed differed from the 2M natural material and was later shown to resemble synthetic 1M material prepared by Yoder and Eugster (Eugster private communication). One of the minerals formed from silica-alumina gel and magnesium hydroxide had an X-ray-diffraction pattern similar to kaolinite, but with a basal spacing of 7.04 Å., suggesting the presence of amesite (Brindley, Oughton and Youell, *Acta Cryst.* 4, 552, give 7.00 Å.; Steinfink and Brunton, *Acta Cryst.* 9, 487, give 7.02 Å.). (Rayner.)

SPECTROCHEMISTRY

Determinations are being made on the general levels of minor elements occurring in selected sedimentary deposits and in soils derived from them, with attention to the distribution of the elements in the different particle-size fractions. Analysis of the liquids used during mechanical fractionation, and of the fractions themselves, showed that losses to solution are usually small. In the soils studied, losses of Ba, Cr, Ga, V and Zr were negligible; this was also so with Sr in non-calcareous soils. Rather greater losses may occur with Co, Cu, Mn, Ni, Pb and Zn.

In a London clay soil the concentrations of Co, Cr, Cu, Fe, Ga, Mn, Ni and V tend to increase down the profile. The bulk of these elements resides in the clay fractions, and their concentrations in this remain fairly constant; but in the sand and silt fractions they increase considerably with depth. The movement of these elements in this soil seems independent of clay movement, and their accumulation at lower levels takes the form of a deposit on the coarser particles and not on the clay. This is confirmed by a strong Fe staining of the sand grains (*cf.* Soil Chemistry, below).

A comparison of levels of organic carbon in specimens of London clay and Lower Lias shales suggests why grey London clay weathers brown and Lias shales do not. Measurements on many specimens of Lias shale showed that in most organic C was well above 1% and in many above 5%. No specimens of grey London clay, however, contained more than 0.5% organic C; furthermore, in a London clay soil, brown clay at 11 feet contained 0.1%. This suggests that where clay or shale contain enough fossil organic matter, reducing

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conditions are maintained, as in the Lias; but the London clay seems to contain too little to keep all the iron in the ferrous form.

Some spectrographic measurements were made on suspected heavy metal contamination in sewage sludge. Several had abnormally high levels of Zn, Cr and Ni, and it seems the trouble these elements caused was aggravated by the exceptionally dry summer increasing their concentration in the residual soil moisture. (Le Riche and Burnett.)

SOIL CHEMISTRY

The effect of soil waterlogging

The ratio of soil to water in a flooded soil profoundly affects the extent to which iron compounds are converted to water-soluble forms. After 2–3 weeks incubation with a large excess of water, the solution may contain amounts of iron in excess of 1% of the weight of the original soil; incubation of the soil at about water-holding capacity converts only a fraction of this amount of iron into a water-extractable form. At capacity, however, extraction with molar ammonium acetate solution at pH 7 removes a further quantity of iron, and yet more is removed at pH 3. The amount removed at pH 3 is commonly the greater part of the total amount rendered soluble, and with some soils the total extractable iron is comparable with the amount dissolved by incubation with a large excess of water. Nickel and cobalt behaved similarly in experiments on soils containing abnormally high concentrations of these elements.

All the iron rendered soluble might be removed from a soil incubated at low water content by prolonged leaching with water, because extraction of field samples of sulphide-free gley soils at pH 3 yields very little iron, but experience with laboratory preparations indicates that, if this is so, the leaching would take a considerable time.

Considerable difficulty was experienced in obtaining field samples of gley soils free from ferrous sulphide, or excessive amounts of reprecipitated ferric oxide, and no completely suitable material has so far been found. Results obtained with the least unsuitable soils indicate that very little of the residual iron is present in the ferrous form.

The mobilisation of minor elements in flooded soils was further studied. The availability of soils containing appreciable amounts of particular minor elements has dictated the choice of elements studied. Mn, Zn, Pb, Ni, Co, Cu, Cr and Mo are mobilised to various extents and, as with iron, whereas some of the solubilised material can be extracted with water, additional amounts can be leached from the incubated soil by a solution of ammonium acetate. Mo behaves differently from the other elements studied, and less is extracted at the lower pH value.

Copper oxide is readily dissolved by aqueous grass extracts, and the process appears to be non-biological; in fact, the presence of appreciable amounts of Cu appears to inhibit microbiological action, and thus largely prevents the solution of other elements. Increasing the amount of fermentable plant material in the reaction mixture greatly decreases the amount of copper mobilised. This anomalous

effect appears to result from the sorption of dissolved Cu on the insoluble fraction of the plant material.

Only little Cr dissolved when soil containing appreciable quantities of chromite were flooded, perhaps because of the refractory nature of chromite rather than of an intrinsic property of chromium.

Reoxidation and drying precipitates the bulk of the mobilised iron, and the minor elements are co-precipitated with the ferric oxide thus formed; however, appreciable quantities of the minor elements may still be extracted by neutral ammonium acetate—in general, more than can be extracted from the original soil. (Bloomfield, Greet and Ng Siew Kee.)