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## Report for 1954

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### Pedology Department

#### A. Muir

A. Muir (1955) *Pedology Department ; Report For 1954*, pp 54 - 59 - DOI:  
<https://doi.org/10.23637/ERADOC-1-76>

## PEDOLOGY DEPARTMENT

A. MUIR

A. Muir visited Nigeria at the invitation of the Colonial Office and the Nigerian Government, and later attended the 2nd Inter-African Soil Conference as a British delegate and the 5th Congress of the International Society of Soil Science, both held in the Belgian Congo. G. Brown and R. Greene-Kelly attended the 3rd International Crystallographic Congress in Paris. D. M. C. MacEwan has been given leave of absence to take up a post in the University of Granada. Mr. S. Singh was awarded the degree of Ph.D. by London University and has now returned to India, and Prof. J. L. White has returned to Purdue University after his year's work in the department. Dr. D. H. Ya'alon of the Hebrew University, Jerusalem is spending one year studying clay mineralogy.

### SOIL MINERALOGY

#### *Nyasaland*

A study of the mineralogical composition of a suite of soils from the Lower Shire River area has been carried out with the object of establishing the derivation of the various soils, and seeing whether the variation in soil pattern is dependent on the type of source rock from which they are derived. Altogether, samples from twenty-one profiles have been subjected to mineralogical analysis in order to determine the probable parent materials, and it has been possible to subdivide the area into several provinces showing common characteristics in the mineral assemblages.

(1) The heavy minerals are characterized by a dominance of hornblende with opaque iron minerals abundant. Biotite, apatite, sphene and epidote are prominent, and with zircon and garnet, form the bulk of the accessory minerals. Minor heavy accessories and the light minerals suggest derivation from hornblendic rocks of the crystalline complex.

(2) The heavy residue contains a very high percentage of opaque minerals, with hornblende as the dominant pyrobole. Garnet and zircon are both very common. Together with the accessories and light minerals, the assemblage suggests a mixture of material of Basement Complex origin with either (*a*) material of sedimentary origin having a high percentage of opaque minerals or (*b*) very weathered material of basaltic origin from which the characteristic pyroxenes and feldspars have been eliminated, leaving a residue of iron oxides.

(3) All the samples in this group show the influence to a greater or lesser extent of the contribution of basaltic material, as is shown by the presence of pyroxenes and ore among the heavy minerals, and labradorite among the light minerals; zeolites and small amounts of volcanic glass also occur.

(4) One profile has been examined in this area: the sands yield only a small quantity of heavy residue, of which opaque minerals make up more than 50 per cent. The high proportion of the "resist-

ant" heavy minerals, a comparative scarcity of pyroboles and a high quartz content suggest an origin from sedimentary deposits.

(5) The sands in this province yield abundant heavy residues (up to 20 per cent by weight), with hornblende and/or garnet the dominant minerals. All these soils are probably of Basement Complex origin. (I. Stephen.)

#### CLAY MINERALOGY

##### *Soil morphology and mineralogy*

A study of thin iron pan soils (cf. Crompton, *J. Soil Sci.* **3** (1952), 277) by mineralogical and chemical methods has been started. Attention is being concentrated on the iron pan itself and the immediately adjacent soil to elucidate the nature of the iron pan and its effect on the mineralogy of adjacent materials. The iron pan consists of several distinguishable layers, and it is hoped to investigate these separately. (G. Brown.)

##### *Dickite*

In collaboration with Dr. F. Smithson, University College of North Wales, the work on dickite has been continued. A paper on the petrography of the dickite sandstones has been published by Dr. Smithson, and the mineralogical part of this work is now being prepared for publication. Detailed examination of carefully purified samples of dickite from the different geological formations has been carried out. (G. Brown.)

##### *Ion exchange studies*

Apart from some studies on the exchange of ions at elevated temperatures in a bomb designed and made in the Department, no further work has been carried out. (R. Greene-Kelly.)

##### *Adsorption by clay minerals*

The vapour-phase isotherm studies on montmorillonite have been continued, particular attention being paid to the influence of the macro-structure of the clay on the isotherms. The results have shown that whereas the sorption of water is little affected, the adsorption of non-polar molecules appears to be restricted to the surface readily available, and hence is markedly altered, depending on whether the clay is in blocks or is finely divided. The influence of temperature on the sorption of water has been studied with a view to investigating the influence of the hysteresis present in the isotherms on the calculated heats of sorption. Whilst the results await the comparison with calorimetrically determined heats, it appears that there is a wide variation in heats determined from the isotherms, depending on whether the adsorption or desorption path is followed.

During the year a high vacuum adsorption calorimeter has been constructed to measure the heats of adsorption and heat capacities of water films on montmorillonite. The present model is capable of operating over the temperature range  $+30^{\circ}$  to  $-80^{\circ}$  C., and is thus able to explore the region in which freezing transitions are reported.

The calorimeter is fully adiabatic, having a shield which is automatically maintained at the same temperature as the calorimeter by a galvanometer-photocell control.

In connection with the kaolin-water studies reported last year a heat-of-wetting calorimeter, using a thermistor as temperature-sensitive element, has been designed. When completed, it is hoped that it will be able to measure heat effects of the order of 1 calorie with an accuracy of a few per cent.

The study of the sorption of aromatic molecules by montmorillonite has been completed, and two papers will shortly be published. The work on the sorption complexes of montmorillonite and saturated ring and chain molecules has been continued, the main effort being directed to resolving the problem of the variation in apparent contact distances between the molecule and the silicate sheet surface with the number of atoms in the molecule.

A detailed study has been completed on the pyridine-montmorillonite complex with a spacing of 23 Å. The results suggested that the molecules were arranged in a layer symmetrically between the silicate sheets and supported on either side by molecules oriented normal to the silicate sheet surface. (R. Greene-Kelly.)

#### *Differential thermal analysis*

In connection with the chapter on the montmorillonite group being prepared for the Differential Thermal Analysis Monograph, a large amount of work has been carried out. A study of the occurrence of a d.t.a. peak between 500° and 600° C. with these minerals has resulted in the recognition that peak temperature is only partly governed by mineralogical composition. The organic complexes of montmorillonite have also been investigated by d.t.a., and the close similarity between the resultant diagrams and those obtained with water and montmorillonite have been striking. A method has been developed for distinguishing cases of chemical from purely physical sorption. (R. Greene-Kelly.)

#### *Study of the Clay-with-flints*

The material called Clay-with-flints is a very variable deposit. In collaboration with Mr. Avery of the Soil Survey a detailed study of a series of soil profiles developed on this material at Great Hampden, Bucks., has been started with the aim of obtaining an understanding of the soils found on it and also if possible of the origin of the various materials composing it. (G. Brown and D. H. Ya'alon.)

#### *Techniques and methods*

An important factor in determining resolution in the Debye-Scherrer powder method is the thickness of the specimen. A method has been devised, using gum arabic as a binder, to make very narrow specimens. A slurry of gum arabic (10 per cent) and the powder to be photographed is made and allowed to dry on a glass slide. From this aggregate, specimens of approximately square cross-section, 0.1 × 0.1 mm. or less, are cut with a razor blade. By rotating the specimen, effects of preferred orientation are overcome. With an ordinary 9-cm.-diameter non-focusing camera, lines whose  $\theta$ 's differ by less than 15 minutes can be resolved, a resolution which compares

well with the results obtained with larger cameras of a semi-focusing type. (G. Brown.)

Work on the experimental gas X-ray tube, mentioned in the Annual Report for 1953, has been continued intermittently. Early in the year it was decided to consolidate the advances made and to place the experimental model in a state of ready availability for practical use. The flexible design of the set was retained, and it has been possible to carry on further researches in an attempt to improve its performance. Though the overall output of the gas set has been improved, efforts to establish a controlled focus have not been as successful as was hoped for. Focal areas of the order of 1 cm. by 1.5 mm. have been obtained by the use of shaped liners placed inside the tube, in some cases without any appreciable diminution of intensity. It has been found, however, that when designed so as to be most effective in focusing the electron stream, the liners are subject to intense bombardment by electrons, causing those made of aluminium and copper to melt, while one containing molybdenum parts began to disintegrate and rendered the tube unusable.

At the same time as the work on focal dimensions was being carried out, a 9-cm. focusing camera based on the Bragg-Brentano principle was constructed in an experimental form. With this camera it is possible in 5 minutes to obtain a well-exposed photograph from a soil clay on the new set. These photographs do not usually contain a full picture such as might be obtained from a flake or powder specimen, since focusing is only effective over a limited angle; but if certain lines only are required, these can be obtained by correct setting of the specimen. A flake specimen on the same set would require about 90 minutes to provide an equally well-exposed photograph. By using suitable shutters on the focusing camera and re-setting the specimen for each shutter, it is possible in three or four settings of 5 minutes each to obtain a complete picture in 20 minutes, as against the 90 minutes in the ordinary 9-cm. camera. The resolution of the lines obtained with the focusing camera can be controlled by altering the width of the collimating slit, greater resolution requiring longer exposures. Over a limited range and with exposures of 10–15 minutes, a resolution slightly better than that obtained with a powder camera is possible, though specimen size is an important factor. The focusing camera can therefore be used for a rough but rapid identification of a material or as a means of quickly examining a few selected lines with some accuracy.

Focusing cameras of the kind described, if they are to possess good resolving power, require a divergent beam of X-rays emanating from a line source 1 mm. or less in width. It was in order to obtain this line source that work on the focal dimensions of the gas set was undertaken. Though these efforts have not been entirely successful, it is thought that further work in this direction might be worth while.

About the middle of the year, the four-window gas set designed by D. M. C. MacEwan and made in 1951 became unusable, and examination showed that the polythene insulator had become badly damaged by bombardment with ions or electrons during use. The opportunity was therefore taken to fit to this set a completely new tube based on the design of the experimental model previously

referred to, using a porcelain insulator. One or two minor modifications were made at the same time and the general lay-out simplified. This new gas tube is now in service with an increased output of X-rays.

An important feature of both the gas tubes now in use is the ease with which they can be dismantled for cleaning or inspection, so that developing troubles can be dealt with before they become really serious. On re-assembly of the tubes, only minor adjustment of individual cameras is necessary, as the target itself can be moved so as to bring all cameras into alignment at the same time. (G. C. Dibley.)

#### *Reaction of molten salts and micas*

An investigation has been carried out of the reactions between molten salts and micas. A preliminary note giving some of the results has appeared in *Nature*. (Prof. J. L. White.)

### SPECTROGRAPHY

#### *Solution spark method*

Work has gone forward during the year in developing the porous-cup technique of spectrographic analysis. This method of analysing solutions was first described in 1948, and has since been fairly widely developed in trace-element work on rocks and soils and in metallurgy. The source of excitation is a 10,000-volt spark, which jumps in a 2-mm. gap between carbon electrodes, of which the upper one is in the form of a cup. The base of the cup is cut to a thickness of 0.5–0.6 mm., and when once wetted is sufficiently porous to allow a slightly acid solution to percolate when struck by the spark. The medium spectrograph is used with this technique.

The method is more suitable for solutions, e.g., soil extracts, than for solids, as inevitable dilution occurs when the latter are dissolved. There are also indications that high concentrations of alkali salts cause difficulties. Nevertheless, the method is highly sensitive, and magnesium can be read at 0.05 p.p.m. with a 30-second burn.

The A.C. spark is much more stable than the D.C. arc, and results within a standard deviation of  $\pm 3$  per cent seem to be well within the possibilities of the method.

At present, working curves are available for magnesium, using the method developed by Dr. R. O. Scott at the Macaulay Institute, and the method will replace Lundegårdh determinations for Mg in routine analysis. Methods are being developed for other major elements to be used in a rapid silicate analysis of small samples. It is also hoped to use it for determination of boron, zinc and copper, all of which are possible by the porous-cup technique and are generally difficult in the D.C. arc. It is hoped that this technique, together with the use of a recently acquired flame photometer, will largely supersede the Lundegårdh method, with a consequent appreciable saving of time. (H. H. Le Riche and A. H. Weir.)

Early in the year a large number of analyses were done for the Botany Department to determine molybdenum and vanadium in plant ashes in connection with Katherine Warington's plant-nutrition work. Subsequent to this, a new technique, permitting greater

speed in such determinations, has been adopted, and applied to the analysis of plant ash for copper, iron, manganese and aluminium. It can also be used for most other elements occurring at more than about 20 p.p.m., with the exception of zinc and boron. (H. H. Le Riche.)

#### *Geochemical studies*

Pressure of other work and staff difficulties have hampered work on the study of trace-element distribution in the Lower Lias formations. A method is being used which permits the direct determination of organic carbon in these highly calcareous and pyritic materials, and evidence so far suggests that the correlation previously reported between the content of Mo, V, Ni and Cu and that of material oxidizable by the dichromate wet oxidation method is, in fact, a correlation with the organic carbon content and not that of sulphur. Further work is now proceeding on this subject. (H. H. Le Riche.)

Numerous soils from Wales were analysed as part of the programme for determining the general distribution of minor elements in soils from Great Britain. Arsenic increased by a factor of  $1\frac{1}{2}$ –2 from the C to A horizon, but the average for the surface samples (about 7 p.p.m.) was decidedly lower than the average for surface samples from various soils in Lancashire (15–20 p.p.m.). (J. R. Butler.)

### SOIL CHEMISTRY

#### *Effects of leaf extracts on soil constituents*

Continuing work on the soil-forming properties of leaf leachates, further evidence for the operation of an inhibiting factor in the reaction between ferric oxide and an aspen-leaf extract has been obtained. Compared with the original extract, the extent of solution-reduction of ferric oxide is decreased by dialysis through cellophane. On the other hand, if the extract is shaken repeatedly with kaolin, the effectiveness of the resulting solution is considerably enhanced. Neither of these treatments is likely to have involved any change other than the removal of certain constituents—small-molecular-weight species in the case of dialysis, and species of large molecular weight by kaolin sorption. It follows from the kaolin experiment that the sorbable compounds in the original extract exert an inhibiting effect in the overall solution-reduction process; the experiment with dialysed extract shows that the low-molecular-weight constituents are the most active in causing either or both solution and reduction.

Essentially similar results have been obtained by treating the extract with ion-exchange resins, although in this case there is a possibility of artefacts being involved. In general, substitution of  $\text{NH}_4^+$  for other cations results in increased solution, while passing the extract through an anion-exchanger ( $\text{CO}_3^{2-}$  form), results in decreased activity.

Several constituents of aspen leaf and Scots pine needle extracts have been identified, but so far it has not been possible to relate these to the action of the extracts on ferric oxide. (C. Bloomfield.)