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Rothamsted Report for 1963

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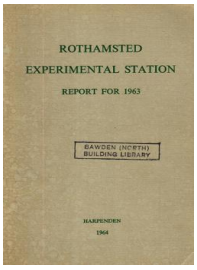

Pedology Department

C. Bloomfield

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PEDOLOGY DEPARTMENT

C. BLOOMFIELD

We regret that I. Stephen resigned because of ill health. D. S. Jenkinson and A. C. D. Newman transferred to the Pedology Department from the Chemistry Department, and H. H. Le Riche from the Soil Survey. J. Catt and H. King were appointed in October, and M. A. Gad, of the Egyptian Geological Survey, arrived in November to spend two years in the Spectrography Section.

The department moved into its new quarters in the West Building extension in May.

C. Bloomfield read a paper at the NATO Palaeoclimates Conference in Durham, and R. Greene-Kelly and A. Weir gave papers at the 1st International Clay Conference in Stockholm; Greene-Kelly acted as a chairman of the section on Clay-Organic Complexes. D. S. Jenkinson gave papers at the FAO Technical Meeting on the use of Isotopes in Soil Organic Matter Studies at Braunschweig and assisted in editing the proceedings.

Mineralogy

Ferrous sulphate hydrates. The cell dimensions for an analysed sample of $FeSO_4.H_2O$ (Fe 32·7, SO_4 56·4%), prepared by the action of concentrated sulphuric acid on ferrous sulphate, were refined by least-squares treatment of indexed X-ray diffraction powder data. This gave lattice constants for a *C*-face centred cell $a = 7{\cdot}087$ Å, $b = 7{\cdot}545$ Å, $c = 7{\cdot}605$ Å, $\beta = 116{\cdot}17°$. These data disagree with those reported by Pistorius (*Bull. Soc. chim. Belg.* (1960), **69**, 570–574). The powder pattern of his material is similar to but clearly different from that reported here, and gave $a = 7{\cdot}123$ Å, $b = 7{\cdot}468$ Å, $c = 7{\cdot}624$ Å, $\beta = 115{\cdot}86°$, with space group $C\ 2/c$. These differences suggest that two similar but distinct phases may exist. Pistorius' material was prepared by dehydration of $FeSO_4.7H_2O$ in an inert atmosphere, but he does not give the chemical composition of his product. (Brown)

Rectorite. Our previous work on rectorite and allevardite (*Rothamsted Report* for 1962, p. 72) showed that these are the same mineral, and established that structurally they consist of interleaved mica-like and montmorillonite-like layers. According to our previous analyses, there were considerable discrepancies between the Na_2O contents of the four samples, and so the Na- and K-saturated forms were analysed again. There are now no such discrepancies, and the latest analyses confirm the identity of rectorite and allevardite, and show that the chemical composition is fully compatible with the proposed structure. (Weir and Brown)

Pyrophyllite. We showed in last year's Report that the intensities calculated for a structure in which the oxygen tetrahedra about silicon atoms were

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twisted 9° from the *a* axis gave a better fit to the observed intensities of X-ray diffraction than those given by previously proposed structures for pyrophyllite. Our data related only to the zero, first- and second-layer lines along the *a* axis, and so we examined the same crystal by Weissenburg photography, for the zero and four higher layer lines, for rotation about the *b* axis. This gave more information, but the intensities of those reflections occurring in both sets of photographs did not agree well. We are trying to find the reason for this and to correct the two sets to enable them to be used together in improving our model of the structure. The disagreement may arise either from differences in the extent of absorption for two settings of the crystal, or from differences in the shapes of the reflections in the *a*- and *b*-axis photographs. MoKα radiation was used in an attempt to minimise absorption effects, but, because of the shorter wavelength dispersion was less and the already diffuse spots merged together. We are attempting to correct for absorption by calculation, but this is time-consuming, and a programme for the computer will have to be written.

It would be an advantage to be able to study a less disordered crystal; we have examined many crystals with this in mind, but with no success. More accurate cell dimensions have been obtained from *a*-axis oscillation and rotation photographs taken with the CuKα radiation and Straumanis film mounting. (Brown and Rayner)

Nitrophosphate fertilisers. Samples of four commercial nitrophosphate fertilisers, and their residues after removing water-soluble and alkaline citrate-soluble components, were examined for the Chemistry Department in connection with an investigation of the agricultural value of nitrophosphate fertilisers (*Proc. Fertil. Soc.* (1963), No. 75, 57–97). The phases were identified and estimates made of their relative abundance. For two nitrophosphates, made by the same process, the one with the poorer performance in field experiments was known to contain much less alkaline citrate-soluble phosphorus. X-ray analysis showed that the less-effective product contained less monetite and more apatite-like material. (Brown with Mattingly, Chemistry Department)

Physical Chemistry

Clay Soils. The single-plate apparatus is unsatisfactory for studying suction curves of clay soils because the difficulty of restricting evaporation necessitates the use of a very thin layer of soil if suction equilibrium over the whole sample is to be achieved. The twin-plate apparatus is more satisfactory, and we have replaced our equipment with five sets of twin-plate units.

We have developed an accurate pyknometer for measuring the bulk densities of soils, and also a high-pressure permeability cell, both of which appear to be satisfactory.

In connection with the study of the movement of water in soils we have been testing field suction equipment and nylon resistance gauges. Preliminary laboratory work confirms reports in the literature of drift in the calibration of resistance units.

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Water in clays. We continued our study of the nature of sorbed water on Na–montmorillonite and found that the specific heat of a monolayer of water is only just below that of liquid water. Pseudo-freezing starts at about −30° C and extends down to considerably lower temperatures. Below −130° C the specific heat curve follows the ice curve, but is always a little above it. (Greene-Kelly and Gallavan)

Release of potassium from mica. In the investigation of the release of potassium by mica minerals, preliminary work was begun on a purified commercial "vermiculite" containing 4% K.

Examination of the starting material by X-ray diffraction showed two sets of irrational *d*(001) reflections: a series based on 14 Å and a series based on 23–24 Å. The material therefore seems to be a mixture of vermiculite and irregularly interstratified biotite–vermiculite. Extraction of potassium from the material with sodium tetraphenyl boron gave a product containing 0·27% K. After calcium saturation it gave an almost rational series of *d*(001) reflections based on 14·8 Å, rather smaller than the spacing expected for Ca–vermiculite (15·1 Å). The cation exchange capacity was determined by two independent methods. Exchangeable Ca extracted by *M*-NaCl amounted to 191 meq/100 g ignited weight, whereas total analysis of the Na-saturated material showed that it contained 194 meq/100 g, which is reasonable agreement. Ultimate analysis of the material before and after extraction of potassium indicated that removing potassium caused small changes in the average composition of the tetrahedral and octahedral layers. Before this work can be extended to well-defined potassium micas the methods used to determine total Al, Mg, Ca and Fe in silicate minerals will need to be critically examined, so that any concurrent changes in chemical composition during potassium release can be estimated accurately. (Newman)

Calculation of mass spectrometer results. A programme was written for calculating the percentage of N-15 in nitrogen from mass spectrometer measurements. Operation of the mass spectrometer for one week involved calculations lasting about 4 hours; these can now be done in 5 minutes by the computer, but punching the teleprinter tape takes about 1½ hours. If the mass spectrometer results were punched on cards these could be used directly on the Orion computer. (Rayner and Newman)

Spectrochemistry

Continuing our study of the location of trace elements in soils, we are attempting to develop an improved method for concentrating lead, molybdenum and zinc, which usually occur in soils below the limits of detection. These elements are normally concentrated by precipitation, but this method is not applicable to soils because of the presence of predominating amounts of iron and aluminium. We are attempting to isolate these elements by the use of an ion-exchange resin, which is ashed and the residue analysed spectrochemically.

The interruption of work caused by our move to new premises provided

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an opportunity to make improvements and adjustments to equipment. The delay caused by this, and the necessary recalibration, has meant that the unit has been out of action for several months. Recalibration is now nearly complete and work is proceeding again. The transfer of the magnesium direct-reading instrument presented no difficulties, and it was soon back in working order. About 2,400 magnesium determinations were made.

A considerable proportion of the section's time was spent on work for the Chemistry Department and for outside organisations, i.e., the Glasshouse Crops Research Institute, Littlehampton, the Equine Research Station, Newmarket, Chemera Research Station, Kuala Lumpur, the North Borneo Department of Agriculture and the British-American Tobacco Co. (Le Riche and Burnett)

Soil Chemistry

Decomposition of plant material in the field. An experiment was started in 1962 on the decomposition of ryegrass, uniformly labelled with carbon-14, in soils of differing pH values, organic matter and clay content. The rate of decomposition under grass was compared with that under bare fallow. The soils were incubated in small lysimeters in the field, from a few of which the drainage was collected. Soils 1–5 and 8 (Table 1) were all from Rothamsted (1 and 2 from Broadbalk, 3, 4, 5 and 8 from Park Grass) and had similar clay contents, although differing widely in pH and organic matter content. Soils 5 and 6 were sandy, 5 being a near neutral soil from Woburn, and soil 6 an acid sandy soil from Bagley Heath.

TABLE 1

Decomposition of labelled ryegrass in soils

Soil number	1	2	3	4	5	6	7	8	8
pH of soil	8·1	7·8	6·9	4·8	3·7	6·2	3·7	7·1	7·1
% organic C in soil	0·97	2·43	4·57	3·87	3·96	1·60	2·86	4·18	4·18
% clay in soil	17·5	18·0	19·7	20·5	20·7	7·5	5·3	19·0	19·0
Plant addition*	Tops	Tops	Tops	Tops	Tops	Tops	Tops	Roots	Roots
Treatment	Bare	Bare	Bare	Bare	Bare	Bare	Bare	Bare	Under grass
% ryegrass addition retained after 1 year†	31·0	32·4	31·3	31·3	42·4	26·7	35·7	27·9	38·5

* 0·125% plant carbon (labelled) added to soil in all cases.
† L.S.D. (5%) = 2·8.

Table 1 gives data on these soils, and on the amount of plant carbon retained after exposure in the field for 12 months: the main conclusions from the first year of the experiment are as follows:

1. For soils with the same clay content (1–5) the retention of carbon is the same between pH's 8·1 and 4·8, but is a third greater at pH 3·7.
2. A change in the organic matter content of the soils from 0·97 to

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4·57% organic carbon does not influence the retention of plant carbon (soils 1–4).

3. The retention of plant carbon by soil 1 (17·5% clay) is significantly greater than that by soil 6 (7·5% clay). The leachate from soil 1 contained 1·6% of the plant carbon originally added to the soil, compared with 3·8% from soil 6, so that the "protective" action of clay is due in part to reduction in the loss of organic material by leaching.

4. The acid sandy soil retained more carbon than the near neutral soil 6, but soil 7 (5·3% clay) retained less than the acid soil 5 (20·7% clay), so that the clay appears to have a "protective" action over the whole pH range studied.

5. The retention of labelled plant carbon was 38% greater in soil growing grass than in the same soil kept bare. This is most probably due to dessication of the soil under growing grass, but other explanations are also possible. This effect may be connected with the accumulation of organic matter under grass, in chernozems, for example.

6. Little decomposition took place during the second six months (October–March) of the experiment; the retention of plant carbon after six months did not differ significantly from that after 12 months. (Jenkinson)

Equations commonly used for predicting the loss of organic matter from soils assume that the amount of humified matter decomposing per year is proportional to the amount present. Our measurements of the rate of decay of tagged plant material suggest that this assumption is not justified. If it is assumed that soil organic matter consists of a continuous range of substances, each of which decays exponentially but at differing rates, then the distribution of rates is the inverse Laplace transform of the decay curve. This inverse transform cannot be calculated directly but, with the help of the Statistics Department, a good approximation to some of our data on the loss of carbon from ryegrass decomposing in a calcareous soil at 25° C was found. The median half life of the plant carbon was 95 days, but 10% of the carbon had a half life longer than 5·8 years. (Rayner and Jenkinson, with Ross, Statistics Department)

Palatability of forest litter to soil fauna. In collaboration with the Entomology Department we are examining oak leaves in an attempt to determine the chemical differences responsible for their different degrees of palatability to soil fauna. Preliminary results indicate significant differences in the proportions of high- and low-molecular-weight polyphenolic compounds in palatable and unpalatable leaves. (King and Bloomfield, with Heath, Entomology Department)

Mobilisation of trace elements in soil. Following work on the mobilisation of trace elements in waterlogged soils described in our Report for 1960, we have done preliminary experiments on the effect of aerobically decomposing plant material in mobilising trace elements. Significant amounts of Fe, Mn, Co, Ni, Cu, Pb and Mo were mobilised under these conditions. (Bloomfield and Pruden)

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Apparatus

Sedimentation analysis of soil using accumulation curves. An attempt was made to improve the sensitivity of Weigner's sedimentation apparatus (*Landw. VersSta.* (1918), **91**, 41–49), by fitting a capacitance transducer cell in the side tube to indicate changes in the hydrostatic pressure of the sedimentation column. Variation of the height of the water column within the cell causes changes in capacitance which are detected by standard electronic techniques.

The capacitance cell consists of a brass tube with a co-axial central wire forming the other electrode, and is part of the tuned circuit of an R.F. oscillator. The oscillator output is passed into a frequency sensitive demodulator, the output of which is amplified and either displayed on a large-scale meter, or used to operate a potentiometric recorder. We have used the first method, plotting the accumulation manually. The method has been checked using fine sand separated by standard sieving techniques into two particle size ranges (200–100 μ) and (100–50 μ); the curves obtained agreed closely with those predicted by theory. (Farrow with Bascomb, Soil Survey)

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