X-ray diffraction experiments on illite and bravaisite.

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Unpublished work carried out in this laboratory several years ago had shown that the first-order basal X-ray reflections of illite from South Wales[1] occurred at a spacing of about 10Å. both with potassium- and calcium-saturated samples. Recently Jackson and others[2] observed that hydrated mica samples having a 10Å. basal spacing when saturated with potassium, showed a 12 to 13Å. spacing after saturation with calcium. They interpreted their results by ascribing to illite a limited capacity for reversible lattice shrinkage and expansion, identical in type with that known for montmorillonite.

In order to study the problem further three illite and two glauconite samples were treated with concentrated calcium chloride solutions for five weeks under frequent renewal of the solution. At the end of the period, and at various intervals in between, samples were washed until free from excess chloride, and their basal spacings were determined by X-ray diffraction. Ordered aggregates[3] were made by evaporation over 45% sulphuric acid (50% relative humidity), and hydrogen passed through acid of the same strength was circulated through the X-ray camera during exposure.

Details of the samples and results are given in table I and illustrated by fig. 1. The 10Å. basal reflection of illite is not a sharp line, but a band of measurable width, and spacings calculated for both edges of the band are given in the table.

Table I. Basal reflections, in Ångström units, of illite and glauconite samples before and after saturation with calcium.

	Before.	After.
Illite, South Wales	9·8–11·0	9·7–11·2Å.
Illite, Grundy County, Illinois (*a*)	9·7–11·4	9·7–12·5
Illite, Vermilion County, Illinois (*a*)	9·7–11·3	9·7–12·3
Fresh glauconite, Chobham Common, Surrey, England (*b*)	9·8–10·9	9·7–10·8
Fine fraction of weathered glauconite, Knaphill brickworks, Surrey, England	9·9–11·8	9·7–10·4 13·5–16·0

(*a*) Material kindly supplied by R. E. Grim.
(*b*) Material kindly supplied by C. O. Harvey.[4]

It may be seen that the illite from South Wales and the fresh glauconite showed no change after the calcium-treatment; two other illite samples (from Illinois)

[1] G. Nagelschmidt and D. Hicks, Min. Mag., 1943, vol. 26, pp. 297–303.

[2] M. L. Jackson and N. N. Hellman, Proc. Soil Sci. Soc. Amer., [1942], vol. 6 (for 1941), pp. 133–145. [M.A. 9–78.] N. N. Hellman, D. G. Aldrich, and M. L. Jackson, ibid., 1943, vol. 7 (for 1942), pp. 194–200. [M.A. 9–78.]

[3] G. Nagelschmidt, Journ. Sci. Instr., 1941, vol. 18, pp. 100–101. [M.A. 8–137.]

[4] C. O. Harvey, Amer. Min., 1943, vol. 28, pp. 541–543. [M.A. 9–83.]

showed a small but distinct broadening of the basal reflection towards larger spacings; and the fine fraction of weathered glauconite showed in addition to the 10Å. band a line at 15Å.

The following explanation is suggested. Illite is not a stable mineral under humid temperate conditions, and the first well-defined product of breakdown is a member of the montmorillonite group. The change is accomplished if all K–O bonds are broken. But there may be intermediate stages where sufficient potas-

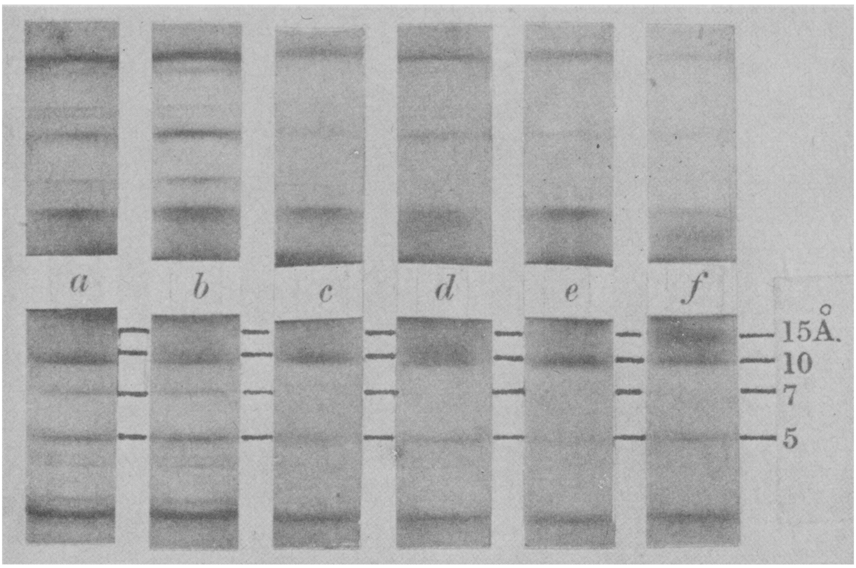

FIG. 1. Aggregate X-ray diagrams, Co-Kα radiation, of: illite from South Wales, (*a*) before, (*b*) after calcium-treatment; illite from Grundy County, Illinois, (*c*) before, (*d*) after calcium-treatment; glauconite, (*e*) fresh, (*f*) weathered, both after calcium-treatment.

sium ions are left in some basal planes to keep neighbouring silicon-oxygen layers together and thus producing the 10Å. spacing; whereas in other planes, after calcium-saturation and the consequent entrance of water, the spacing is 15Å. This interstratification of 10 and 15Å. packets can be of different types. If it is of the 10–15–10–15–10, &c. type an intermediate band may appear, as apparently observed by Jackson. A mixture of such material with unaltered illite would give the result shown in fig. 1*d* (calcium-treated Grundy County illite). If there are large 10–10–10– and 15–15–15–, &c. packets two lines will occur as in fig. 1*f*.

Most of the changes observed on calcium-saturation were apparent after the shortest time interval tested, and the prolonged treatment seemed to have little, if any, further effect, except possibly with the illite from Grundy County. Presumably the weathered glauconite contained, apart from glauconite, a member of the montmorillonite group, and the calcium-treatment only facilitated its identification. To what extent illite samples differ in resistivity to calcium chloride or similar reagents remains to be seen. The few data reported above suggest that illite from shales and fresh glauconite (7·6% K_2O) may be more resistant than illite from unconsolidated clays.

Obviously quantitative X-ray measurements combined with water and potash determinations would be required for a more detailed analysis, but in the writer's opinion it should be accepted, almost one might say by definition, that the basal spacing of illite is a multiple of 10Å., irrespective of the nature of the exchangeable ion. Inasmuch as observed X-ray diffraction effects differ, the materials should be classed, as suggested by Hendricks,[1] as mixed illite-montmorillonite structures.

According to Fleischer[2], bravaisite may be the specific mineral species in illites. Through the courtesy of F. A. Bannister a sample of this mineral from the type locality, Noyant, Moulins, Allier, France, was obtained from the British Museum collection. Basal reflections of calcium-saturated material, *d* 11·2–12·9Å., were found to be distinctly larger than those of potassium-saturated material, *d* 10·2–11·3Å. Small samples brought to equilibrium by drying over air of 50% relative humidity at 20° C. and subsequently heated to 130° C. lost 6·3 and 4·1% of the air-dry weight for calcium- and potassium-saturated samples. This difference is far greater than that found in parallel experiments with illite from South Wales. The bravaisite, while showing X-ray diffraction data similar to those described by Jackson and others (loc. cit.) would, according to the comments made above, be classed as a mixed illite-montmorillonite structure.

[1] S. B. Hendricks and L. T. Alexander, Soil Sci., 1939, vol. 48, pp. 257–272. [M.A. 9–78.]
[2] M. Fleischer, Amer. Min., 1943, vol. 28, p. 470.