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MINERAL HETEROGENEITY IN "GLAUCONITE" PELLETS*

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Abstract

The term "glauconite" is currently being used with a dual connotation. Originally coined (Gr. *glaukos*=bluish-green) as a description for a blue-green micaceous mineral, this word is now widely used as a rock term for small, spherical, sometimes green earthy pellets. By x-ray diffraction, those pellets can be grouped in four general mineralogical classes, only one of which has the diffraction properties of the mineral glauconite. These differences are not reflected in the size, shape, or color of the pellets.

In addition to unique x-ray properties, the mineral glauconite has been characterized by a lath-shaped micromorphology and by a restricted chemical formula which requires that at least two-thirds of all possible potassium positions be occupied. A separate classification has been established for certain potassium-deficient structures on an order-disorder basis. In general, this division can be correlated with Yoder and Eugster's (1955) 1 M-1 Md division of low grade mica structures.

INTRODUCTION

The term "glauconite" has been used interchangeably for many years to describe a specific micaceous mineral as well as a particular morphological form. This dual usage results from the mineral species being found frequently in the form of small rounded greenish pellets. Careful mineralogical investigation of the pellets by petrographic and *x*-ray methods can readily distinguish the mineral glauconite from other similarly appearing materials (i.e. clay minerals). Geologists, however, who work in the field and whose studies of outcropping rocks and well samples are usually confined to binocular microscope investigation, rely essentially upon outward appearance for identification. This tends to pre-empt the morphological interpretation of the term glauconite.

In this manner, the designation "glauconite," originally defined as a mineral name, has become a "wastebasket" into which is placed almost any pellet that cannot be identified as a distinct mineral species of another kind, for instance, siderite or fluorapatite. X-ray work has shown rather conclusively that the term "glauconite" as it is commonly used is more often a generalization or, at best, a rock term rather than a precise mineral designation. It has been found that many materials termed "glauconite" in conversation and in the literature are actually composed of micaceous, chloritic, or montmorillonitic clay minerals.

One of the first descriptions of this material was by Alexander von Humboldt in 1823 [Schneider, Hyrum (1927)] who used the term grün erde to denote the occurrence of the green earthy material in sandstones and limestones throughout Europe. Equivalent designations of terre

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verte and green sand were made in other countries. Keferstein adopted the term "glauconit" (subsequently glauconite) in 1828 from the Greek glaucos = bluish-green and ite = resembling [Brown, R. W. (1954)]. It is not clear if the term at that time was a mineralogic or morphologic description, but the "ite" suffix by subsequent convention pressured for mineral interpretation. No attempt will be made to trace the history of glauconite literature in this report as this has been done on several previous occasions. For extensive bibliographies, references should be made to Schneider (1927), Hadding (1932), Gruner (1935), Steenhuis (1937-39), Hendricks and Ross (1941), Sabatier (1949), Smulikowski (1954), and Cloud (1955). Except where otherwise noted, the author of this report considers the term "glauconite" a morphological description because of the obvious inconsistency of defining the mineral glauconite as mineralogically heterogeneous.

Orgin and Composition

Classically, the mineral glauconite is a hydrous, iron-alumino silicate quite similar to members of the illite clay group. Actually it may be a highly ferriferous end-member composition within that group. Distinguishing chemical features include a K_2O content of 7–8 per cent and total iron of 20–25 per cent in which the ferric iron has been reported to exceed the ferrous by a multiple of 3 to 9. The average ferric-ferrous ratio in 34 sample analyses recorded in the literature was found to be 7.2 [Hendricks and Ross (1941); Hadding (1932)].

Important environmental interpretations have been drawn from this rather restricted range of ferric-ferrous ratios under the assumption that they reflect the redox potential (capacity to reduce or oxidize a system) in the environment where the glauconite formed. The particular range of values usually displayed by glauconite suggests a semioxidizing to semireducing environment. Several theories of origin—(1) fecal pellet conversion, (2) foraminiferal cavity-filling, (3) alteration of biotites, and (4) clay pellet agglomeration on the sea floor in response to undulating currents—can all be resolved by establishing the conditions of origin as requiring, simply, the silicate lattice, supplies of both potassium and iron, and a favorable oxidation potential.

An explanation for the association of glauconite with marine bioforms becomes apparent under these conditions. Decaying organic material (from dead animals or fecal mucous) creates a local reducing environment which sufficiently counteracts the over-all oxidizing marine environment to produce the semioxidizing conditions necessary to glauconite formation.

Alteration to glauconite of various silicates not in close association

with decaying animal matter would be accomplished in a depositional area of natural semioxidizing conditions such as restricted basins or lagoons. Keller (1956) has recently described a green pelletal material from the Morrison Fm., Wyoming, which appears to be of lacustrine origin. Compared to most marine glauconites this material is highly aluminous, resembling the illites.

Theoretically, if there were only one set of environmental conditions pertinent to glauconite formation, its chemical and physical characteristics would be expected to be consistent. Variations in chemical analysis, and heterogeneity in mineralogical nature which has been recently noted [Burst (1952, 1953, 1954); Ehlmann (1954)] are probably due to different chemical environments. The most difficult contribution to evaluate is the iron ion. Most of the ancient glauconites which can be reliably designated as marine are highly ferriferous, suggesting that certain bottom dwelling organisms concentrate iron through digestive processes and deposit it with their fecal pellets.

Tarr (1920) suggested that the iron in glauconites is thrown down by oxidation. Still another suggestion maintains that glauconite is secondary after the high iron biotite lattice and does not require ferriferous contributions from the ocean waters. Although Galliher (1939) has found evidence of this with respect to the weathering of large biotite grains, the formation of glauconite by this method from heterogeneous marine sediments does not seem feasible. As most of these deep marine pellets appear to be converted fecal material, the suggestion is based on a selection process entailing an organism whose dietary habits enable the unerring segregation of biotitic and muscovitic lattices. Greenish colored fecal pellets found in the white sand beach at Gulf Beach, Florida showed little relation to either biotite or muscovite. Other fecal materials and Recent glauconites have been noted deficient in both iron and potassium.

Smulikowski (1954) agrees with a potassium deficiency in Recent and Tertiary glauconites; however, he often notes an excess of ferric iron. According to him, the formation of glauconite in Recent seas probably occurs at a stronger, on the average, concentration of ferric iron, than in seas of previous geological epochs. At the same time less interlayer cations (chiefly potassium) are now adsorbed by glauconite in the course of formation, than was generally possible in older geological epochs, especially the Paleozoic epoch. This would mean that the geochemical conditions at the sea bottom, in places favorable for glauconite formation, have undergone during the whole geological history a continual evolution in the direction of a growing concentration of iron and decreasing concentration of potassium.

Conway (1942) also notes the deficiency of potassium in clays of Re-

cent and Tertiary ages, as well as in the sea water of these epochs. Actually, he plots a potassium content versus geologic time graph showing this effect.

The present author suggests that both iron and potassium experience oriented migration to favorable concentration centers within defective three-layer clay lattices. The mobility of the iron atom is well recognized through its occurrence in sedimentary deposits as siderite, pyrite, and marcasite concentrations. Its ability to segregate is demonstrated adequately by the ironstone concretion frequently found in essentially ironfree fire clays.

PREPARATION OF ORIENTED DIFFRACTION SLIDES

Pelletal purity is essential to glauconite classification on a mineral basis. The extreme difficulty involved in extracting pure glauconite

TABLE 1. "GLAUCONITE" PELLET SIZES

Average of 10	pellets in	milligrams
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Celadonite (Italy)	1.677	Carrizo Fm. (Texas)	0.056
Navesink Fm. (N.J.)	0.477	Wilcox Fm. (La.)	0.044
Franconia Fm. (Wis.)	0.273	Wilcox Fm. (Texas)	0.037
Winona Fm. (Ala.)	0.211	Gatchell S.S. (Calif.)	0.031
Weches Fm. (Texas)	0.085	Rio Negro Fm. (Ven.)	0.009
Bashi Fm. (Ala.)	0.072	Tibu Fm. (Ven.)	0.009
Reklaw Fm. (Texas)	0.056	Pierson Glauc. (Texas)	0.008

pellets from sediments often results in imperfect separation which, in turn, leads to x-ray and chemical analyses of materials which are essentially glauconite pellets, but which have significant, although minor matrix impurities. It has been found absolutely necessary to hand-pick every pellet used for analysis. Particle weights listed in Table 1 are indicative of the difficulty involved.

To cope with this time-consuming task, micromethods for both chemical and x-ray oriented slide techniques were used. Both analyses can be accomplished with approximately 50 milligrams of material. SiO_2 , R_2O_3 , and water loss are measured gravimetrically, MgO and CaO titrametrically, K_2O and Na₂O spectrochemically, and ferrous and ferric iron colorimetrically.

An oriented clay slide is made with about five milligrams of material by introducing it into a miniature sedimentation column mounted directly on a microscope slide. After settling, excess liquid is decanted and the column removed from the slide leaving the one-dimensional "crystal" for irradiation.

X-RAY CLASSIFICATION

Neither size, nor shape, nor color can be used to distinguish pellets of the mineral glauconite from the many pellets which qualify as glauconite on a morphological basis. Materials of different sizes, shapes, and colors can have the same mineralogy, whereas other materials which are apparently similar morphologically have been found dissimilar mineralogically.

In this report, therefore, the small, rounded, green pellets, defined from a morphological point of view as glauconites are being reclassified into four major groups on the basis of x-ray analysis. The first group contains those materials which possess the structural properties generally attributed to the mineral glauconite. These properties are represented in Fig. 1a as three basal diffraction peaks of the micaceous 10-angstrom lattice. The relative intensities of the 10-, 5-, and 3.3-angstrom lines as well as their sharp symmetrical appearances are the most obvious diagnostic criteria in this type of analysis. The second group is also micaceous and monomineralic. The peaks, however (Fig 1b), are subdued, displaying broad bases and asymmetric sides. This suggests a less rigorous structural scheme and possibly a more moderate impress of formational environment.

Interlayered clay-mineral pellets are accommodated in a third group (Fig. 2) without differentiation as to the nature of the layer intercalation. It was found that most glauconite pellet forms contain interlayered material to some degree. A fourth group (Fig. 3) has been established for the many instances of mineral mixture found in pellet form. Most frequent combinations are illite with montmorillonite and illite with chlorite. No separate classification has been established for pellets containing minerals which could be classified as impurities (i.e., quartz, calcite).

Recently Levinson (1955) and Yoder and Eugster (1955) have shown that mica polymorphic classifications can be extended to relatively disorganized structures strikingly similar to glauconite lattices. These are the 1 M and 1 Md polymorphs obtained experimentally at low temperatures and generally accepted as being the most likely stability fields for sedimentary micas.

A survey of the glauconites in random powder mounts proved that the so-called ordered and disordered groupings can be similarly made on the basis of 1 M and 1 Md classification. Each of the glauconitic materials can be classified as single-layer monoclinic mica with the degree of singlelayer crystallinity varying between 1 Md and 1 M. Franconia glauconite and Navesink glauconite (New Jersey greensand) appear to have 1 M structures (Fig. 4). There are insufficient reflections to entirely satisfy





the 1 M data such as listed by Yoder and Eugster (1955); however, more lines are present than the 1 Md classification would permit. Conversely, Pierson glauconite seems to be fairly representative of the 1 Md type mica structure. Basal reflections are extremely low. The 4.48-Å region is smeared out in the asymmetry typically indicative of disorder and the 2.55to 2.58-Å reflections appear more or less broad and diffuse.

The 1 Md or disordered lattice is not readily apparent in the Bashi glauconite which had been classified as disordered by virtue of the diffraction relationships observed on patterns resulting from oriented flake-type sample mounts. The Bashi, however, is noted as the least



FIG. 2. X-ray diffractograms of interlayered glauconites.

disordered of the disorder type glauconites; therefore, it is quite possible that its pattern would more resemble the ordered lattice type than other materials farther down on the disorder scale.

Estimates of order-disorder relationships are not significantly different, regardless of which type of sample mount is used—oriented aggregate or random powder. The dividing line between the ordered and disordered structures was however, more difficult to approximate if only the random powder patterns were taken into consideration and may be more or less obscured by impurities such as the small calcite accessory in the Burditt specimen.

General correlation between the two types of sample mounts was



FIG. 3. X-ray diffractograms of mixed-mineral glauconites.

made possible due to imperfect orientation of clay platelets. The oriented flake sample is designed to eliminate (hkl)'s and consequently, to eliminate the characteristics of randomness on x-ray diffraction patterns. Perfect orientation of the flakes, however, is not realized, and a few of the disoriented layers are in a position to reflect (hkl)'s even though the sample has been oriented for reflections of basal spacings only.

The fact that the 4.48-Å reflection can be eliminated in the so-called ordered glauconites by orientation and, seemingly, cannot be eliminated from the patterns in the disordered glauconites suggests that, although the orientation in each of the sample types is comparable, disorder may provoke some departure from planarity in the grossly oriented structures.



FIG. 4. Mica polymorphs in glauconite.

CHEMICAL CLASSIFICATION

Obtaining chemical analyses of glauconitic materials can become a very difficult task owing to problems of availability, concentration, and purity. Except for those well-documented and classical glauconite exposures which yield the materials frequently described and redescribed in the literature, few glauconitic beds contain pellets in sufficient proportion to allow the accumulation of the quantities necessary for analytical study by any easy method. Large quantities of sample material are usually required to produce a small quantity of glauconite pellets. In the petroleum industry, however, glauconitic material about which mineralogic information is desired must be extracted from the usually small downhole sample recoveries. This necessitates the adaptation of microanalyses, both x-ray and chemical, to the glauconite work.

	1	2	3	4	5	6	7	8	9	10
SiO ₂	53.22	35.67	48.0	52.7	48.66	47.1	49.0	51.7	51.5	47.5
Al_2O_3	21.19	14.56	10.7	13.7	8.46	11.5	8.4	10.9	23.6	15.45
Fe_2O_3	1.22	3.03	18.0	9.5	18.80	16.00	20.0	14.5	10.0	13.4
FeO		23.23	3.9	5.0	3.98	8.00	2.4	4.3	3.7	3.15
MgO	6.02	9.24	3.0	3.5	3.56	3.6	4.0	4.5	2.00	2.9
CaO		1.13	3.0	.3	. 62	1.3	.3	.000	.06	1.2
K_2O	11.20	8.06	7.8	8.2	8.31	8.2	7.0	6.15	4.8	6.4
Na ₂ O	.34	.49	.18	.1	0.00	.2	.18	avg. .23	.62	.16
H ₂ O ¹⁰⁰⁰ 100 C			7.4	6.8	6.56	6.44	7.5	6.9	4.7	11.1
Total			101.98	99.80	99.80	102.34	98.78	99.18	100.98	101.26

TABLE 2. GLAUCONITE C	HEMICAL ANALYSES	IN WEIGHT	Per Cent
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1. Alurgite [Grim et al. (1937)].

2. Biotite [Gruner (1935)].

3. Navesink Fm., N. J. Greensand, Cretaceous.

4. Ceratopyge Sandstone, Sweden, Ordovician.

5. Bonne Terre Fm., Missouri, Cambrian [Hendricks and Ross (1941)].

6. Franconia Fm., Wisconsin, Cambrian.

7. Bashi Fm., Alabama, Eocene.

8. Pierson Fm., Texas, Eocene.

9. "Subsurface," California, Eocene.

10. Burditt Marl, Texas, Upper Cretaceous.

A combination of general microchemical techniques for the components (Table 2) silicon, R_2O_3 , magnesium, and calcium, spectrochemical techniques for sodium and potassium, and colorimetric techniques for ferrous and ferric iron has been developed for use on samples of less than 50 mg. and has been used to construct the chemical formulas shown in Table 3 for various of the glauconites studied. The formulas are based on an idealized unit cell of 24 oxygen atoms which can accommodate, after allowances for hydrogen, 44 cationic valences. These must be distributed among the materials contained in the elemental analysis to effect neutrality [Kelley (1945)]. It has been found that separating the potassium

atomic equivalence from the so-called exchange ions (sodium and calcium) when writing the formulas provides a convenient method for the comparison of x-ray and chemical data.

It is interesting to note in comparing Table 3 with Fig. 1 that those materials which display the x-ray diffraction patterns of well-ordered singlelayer micaceous materials consistently contain more potassium than do those materials which possess disordered mica structures. When the potassium atom equivalent falls below approximately 1.4 per unit cell,

Bonne Terre Fm., Missouri, Cambrian K1.58(Ca)0.10(Alo 70Fe211/Fe049Mg0.82)(Si7.22Alo 78)O20(OH)4 Franconia Fm., Wisconsin, Cambrian Ordered Glauconites K1 52(Na, Ca) 0.27(Alo 81Fe1,74Fe0,97Mg0.79)(Si6 85Al1 15)O20(OH)4 Ceratopyge Fm., Sweden, Ordovician K1 48(Na, Ca) 0.06(Al1 74Fe1 02Fe0 59Mg0 77)(Si7 47Al0 53)O20(OH)4 Navesink Fm., New Jersey, Cretaceous K_{1.43}(Na, Ca)_{0.51}(Al_{0.81}Fe'''₁₉₇Fe''_{1.97}Mg_{0.66})(Si_{6.99}Al_{1.01})O₂₀(OH)₄ Bashi Fm., Mississippi, Eocene $K_{1,32}(Na, Ca)_{0,10}(Al_{0,74}Fe_{2,53}'Fe_{0,30}'Mg_{0,89})(Si_{7,28}Al_{0,72})O_{20}(OH_4)$ Burditt Fm., Texas, Upper Cretaceous Disordered Glauconites K1,20(Na)0.09(Al1.66Fe1.47Fe0.40Mg0.62)(Si6.98Al1.02)O20(OH)4 Pierson Fm., Texas, Eocene K_{1.12}(Na)_{0.05}(Al_{1.25}Fe_{1.58}Fe_{0.52}Mg_{0.95})(Si_{7.41}Al_{0.59})O₂₀(OH)₄ "Subsurface," California, Eocene K_{0.81}(Na, Ca)_{0.17}(Al_{2.53}Fe¹_{1.05}Fe¹_{0.46}Mg_{0.41})(Si_{6.84}Al_{1.16})O₂₀(OH)₄

TABLE 3. CALCULATED FORMULAS FOR ORDERED AND DISORDERED GLAUCONITES

ordered stacking becomes less apparent owing to the decreased binding power normally supplied to the lattice by potassium. True randomness of the montmorillonite stacking type becomes evident in the low potash glauconites with the appearance of the aforementioned 4.48-Å reflection signifying a two-dimensional diffractor. Apparently, disordering begins when fewer than two out of every three possible potassium positions are filled. Peak broadening and asymmetry result.

From these analyses, it would seem that Bonne Terre glauconite and Franconia glauconite represent an end-point mineral composition within

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the mica group to which the mineral name glauconite should be applied. Retreating from this end-point in gradual steps of diminishing potassium content are materials which less and less resemble glauconites and more and more resemble montmorillonites. That this change is transitional over a broad range of potassium values suggests that the potassium fixation is limited not by the capacity of the structure for such a phenomenon, but by the capacity of the depositional medium to supply potassium to the structure.

All of the materials are clearly dioctahedral. The value of cationic equivalence in the octahedral layer is surprisingly similar in the Franconia, Ceratopyge, and Bonne Terre. An excess of octahedral aluminum and a smaller ferric-to-ferrous iron ratio in the Ceratopyge differentiates it from the other three mineral representatives.

It appears that the potassium deficiencies of the clay minerals (micatype) may be direct measures of impurity, assuming that mineral interlayering is considered an impurity. Other impurities, such as quartz or calcite, must be assessed by the observation of abnormal SiO₂ and CaO values. Kelley (1945), clearly established the conditions necessary to correct formula calculation: (1) It must be known that the sample is composed of material of a given lattice type; (2) the purity of the sample must be assured; (3) the chemical analysis must be reasonably accurate.

The designation of a certain degree of lattice randomness evidenced by its x-ray diffraction properties, as a particular glauconite type (i.e. disordered) may, of course, be questioned. The designation of endmembers in isomorphous series is often difficult, and this particular composition may not be a true end-member. However, its frequent repetition among the many glauconites examined seems significant. It may quite possibly be a reflection of a particular chemical environment or set of chemical processes that frequently lead to the formation of a particular glauconite type.

The classification (see Fig. 5) of green micaceous pellets on the basis of potassium content has been used in an attempt to construct a parallelism between the poorly ordered high aluminum micas (illites) and the poorly ordered high iron micas (glauconites). Equivalent genetic stages have been designated for illite and glauconite; degraded illite and disordered glauconite; and mixed-layer clays and mixed-layer glauconites. All six stages are suggested as intermediate between true micas and true swelling clays. Biotite is included because of its high iron content and because direct observation of its conversion to glauconite has been reported [Galliher (1938)]. This change, however, requires the transformation of a trioctahedral lattice to low order dioctahedral.

Smulikowski (1954) divided glauconites in a somewhat similar manner,

but reserved two names; illites and pholidoides for the highly aluminous materials. For instance, the Ceratopyge material described herein, displaying an excess of aluminum over ferric iron in octahedral coordination, a loss of potassium, and a total octahedral cationic equivalence just slightly above four would, under Smulikowski's system, be classified as illite. To him, pholidoides are similar to illites in having excess aluminum over magnesium in octahedral coordination, but differ in that the



K[†]NUMBERS=ATOM EQUIVALENTS OF POTASSIUM PER LATTICE UNIT

FIG. 5. Suggested diagenetic relationships in micas.

total octahedral cationic equivalence is considerable above 4 (by as much as 20 per cent). The differentiation, therefore, is made on a "seat of fixation charge" basis.

MICROMORPHOLOGY

Electron micrograph monitoring of glauconite specimens provided an interesting parameter of glauconite mineralogy (Fig. 6). The wellordered, high-potassium materials appeared as lath-shaped booklets. It further appeared that the best-developed laths occurred in Franconia and Bonne Terre materials where the individual rectangular plates displayed length-to-width ratios of about 5 to 1. This unique morphology



FIG. 6. Electron photomicrographs of well-ordered glauconites.

- 1. Navesink Fm., New Jersey, Cretaceous (×13,000).
- 2. Franconia Fm., Wisconsin, Cambrian (×11,000).
- 3. Ceratopyge sandstone, Sweden, Ordivician (×13,000).
- 4. Bonne Terre dolomite, Missouri, Cambrian (×10,000).

is another useful argument for the establishment of Bonne Terre and Franconia glauconites as type minerals.

Disordered glauconites and mixed-mineral pellets such as shown in Fig. 7 tend toward equilateral plates. Bashi glauconite, which has already been cited as resembling the ordered glauconites, shows a few elongated plates.

Conclusions

Students of glauconite have usually approached the problem with one of two different viewpoints. One group, usually field geologists or others making macroscopic or binocular microscopic examinations, describe



- FIG. 7. Electron photomicrographs of disordered glauconites.
 - 1. Gatchell sandstone, California, Eocene ($\times 20,000$).
 - 2. Weches Fm., Texas, Eocene (×11,000).
 - 3. Bashi Fm., Mississippi, Eocene (×13,000).
 - 4. Pierson Fm., Texas, Eocene (×11,000).

all green, earthy pellet materials as glauconite, obviously a gross classification based on external appearance. The other, usually composed of laboratory analysts, confine the designation to monomineralic micaceous materials of slightly varying chemical composition and prescribe an average formula for the ideal mineral material. In this case, the description is a mineral term.

In this report, the earthy green pellets are divided into four groups by x-ray diffraction; one is a mineral genus and three are rock categories. The highest rank is restricted to materials which apparently approach the rigorous qualifications of specific mineral classification. Within this

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group, two materials, Franconia glauconite and Bonne Terre glauconite; are proposed as representative mineral types. These two materials may therefore be termed neotypes for glauconite. This group possesses unique chemical properties and unique disaggregate morphologies in addition to selective diffraction properties.

Two of the three remaining groups were established for glauconitic materials which apparently bear genetic relationships to the mineral type group, but differ somewhat in potassium content. The highest order of these two groups is considered submineralic because, although its slight potassium deficiency indicates interlayering, the repeated occurrence of this particular degree of interlayering suggests a classification higher than the randomness implied by a rock term. The other group of considerably potassium-deficient three-layer silicate lattices produces poor diffraction patterns and often displays swelling properties.

The fourth category is a designation for green colored clay pellets containing two or more argillaceous minerals.

The reclassification into four specific groups of heterogeneous materials previously described by a single name and often considered mineral equivalents can be used to advantage in stratigraphic and environmental interpretations. It is deemed necessary that "glauconite" designations and classifications be made with the assistance of x-ray diffraction just as is required by other clay-mineral identifications. Morphological descriptions which result from hand-specimen examinations often conceal significant mineral characteristics.

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