

ILLITE DIAGENESIS A QUARTER CENTURY AFTER HOWER ET AL.

Monday June 21, 2004

Illite Diagenesis a Quarter Century After Hower et al. I	
1:20 – 2:00p	JOHN HOWER WAS RIGHT: ILLITE-SMECTITE AS A PALEOGEOTHERMOMETER. Jan Srodon Institute of Geological Sciences PAN, Senacka 1, 31002 Krakow, Poland
2:00 – 2:20p	THE FATE OF SMECTITE BEFORE BECOMING ILLITE IN BENTONITE EXPERIMENTS. A. Bauer ^{*1} , E. Ferrage ² , K. Emerich ³ , and B. Velde ⁴ ¹ FZK-INE, PO Box 3640, 76021 Karlsruhe, Germany, ² Environmental Geochemistry Group, L.G.I.T. - Maison des Géosciences, Université J. Fourier, B.P. 53, 38041 Grenoble Cedex 9, France ³ FZK-ITC-WGT, PO Box 3640, 76021 Karlsruhe, Germany; ⁴ Laboratoire de Géologie, de l'Ecole Normale Supérieure de Paris, 24 rue Lhomond, 75005 Paris
2:20 – 2:40p	THE ROLE OF ILLITE AND I-S NEOMINERALIZATION IN FAULT ZONES. J.G. Solum [*] , and B. A. van der Pluijm University of Michigan, Department of Geological Sciences, 2534 C.C. Little Bldg, Ann Arbor, MI 48109-1063
2:40 – 3:00p	AN INTERPRETATION OF OCTAHEDRAL ORDERING AND SITE OCCUPANCIES OF Fe²⁺ IN MUSCOVITE AND ILLITE. Gary S. Collins ^{*1} , and Philip E. Rosenberg ² ¹ Department of Physics and ² Department of Geology, Washington State University, Pullman, WA 99164
Illite Diagenesis a Quarter Century After Hower et al. II	
3:20 – 4:00p	ILLITIZATION AS A CRYSTAL GROWTH PROCESS. Dennis D. Eberl U.S. Geological Survey, 3215 Marine St., Suite E-127, Boulder, CO 80303
4:00 – 4:20p	ILLITE POLYTYPES AND DIAGENESIS SINCE HOWER ET AL. 1976. Georg Grathoff ^{*1} ; Igor M. Gorokhov ² , and Agnes Impiccini ³ ¹ Department of Geology, Portland State University, Portland, Oregon, U.S.A.; ² Institute of Precambrian Geology and Geochronology, RAS, St. Petersburg, Russia; ³ Universidad Nacional del Comahue, Facultad de Ingeniería, CIMAR. Buenos aires 1400, 8300, Neuquén, Argentina
4:20 – 4:40p	STABLE ISOTOPIC EVIDENCE FOR METASTABLE ILLITE GROWTH IN A MATRIX OF NON-EQUILIBRIUM SMECTITE. Lynda B. Williams [*] , and Richard L. Hervig Department of Geological Sciences, Arizona State University, Tempe, AZ 85287-1704
4:40 – 5:00p	SMECTITE ILLITIZATION IN ORGANIC RICH SHALE OFFSHORE MID-NORWAY. Marianne Bruvoll [*] , Jens S. Jahren, and Per Aagaard Department of Geosciences, University of Oslo, P.O. Box 1047, N-0316 Oslo, Norway

MONDAY JUNE 21, 2004

ILLITE DIAGENESIS A QUARTER CENTURY AFTER HOWER ET AL. I

1:20PM

JOHN HOWER WAS RIGHT: ILLITE-SMECTITE AS A PALEOGEOTHERMOMETER

Jan Srodon

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Fifty years have passed since Kubler and Weaver first observed that 001 XRD peak of illite changes systematically with depth and ascribed this change to improving “crystallinity” at higher temperatures. In the late sixties the nature of this change was described in terms of layer ratio and ordering in mixed-layer illite-smectite and the techniques for measuring this ratio were developed. In 1979 Hoffman and Hower first proposed to use % smectite (%S) in illite-smectite in shales as a paleogeothermometer. Based on the then available field evidence they suggested that the transition from random to ordered interstratification takes place at about 100°C and from the nearest-neighbor to longer range ordering at about 170°C. In the following years some investigators followed this approach but many criticized it, proposing instead various kinetic expressions, relating %S to a number of factors besides temperature (time, chemistry of pore water, type of ordering, chemistry of original smectite).

Despite very abundant literature on the smectite illitization in shales accumulated since Hower’s time, its validity for solving this controversy remains limited for two reasons:

- 1) the %S measurement in shales is difficult and the numbers have to be examined critically before using them;
- 2) the burial history of investigated section often is not reported, so only the present-day temperatures and not the maximum paleotemperatures are available.

The examination of the published data, taking these two aspects into account, makes the present author believe that John Hower was right. Particularly convincing is the evidence of Price and McDowell (*Clays Clay Miner.* 41:134-147, 1993), who found randomly interstratified clays in Proterozoic shales from the Lake Superior basin, which have never been deeply buried. The %S profile of the East Slovak Basin (Sucha et al., *Clay Miner.* 28:243-253, 1993) is used in the following studies to calibrate paleotemperatures.

Three regional studies of the present author and his coworkers seem to support Hower’s paleogeothermometer and suggest that it is more reliable than widely used organic indices:

- 1) The Cambrian clays of Estonia, which have never been buried more than 1000 m contain ca. 20%S illite-smectite, and this occurrence was used as an argument in favor of low-temperature time-dependent illitization. Organic indices support this conclusion. However, K-Ar dating of illite-smectite from Estonian bentonites revealed short-lasting illitization event, coincident with the Caledonian orogeny (ca. 390-400 Ma).
- 2) The Carboniferous shales of the Upper Silesia Coal Basin contain $R > 0$ illite-smectite at the present-day erosional surface, which is inconsistent with R_0 data for never deeply buried E part of the basin. K-Ar dating indicates Cretaceous heating event in the E part and the apatite fission track (AFT) dating supports IS against R_0 evaluation of the paleotemperatures ($>125^\circ\text{C}$).
- 3) %S in the Oligocene shales of Podhale flysh basin evolve gradually NW to SE from >60 to $<15\%$ S. $110-120^\circ\text{C}$ paleotemperature isoline based on %S is coincident with the line of total resetting of the detrital AFT ages (125°C). R_0 numbers are erroneous because of overwhelming detrital vitrinite contamination.

THE FATE OF SMECTITE BEFORE BECOMING ILLITE IN BENTONITE EXPERIMENTS

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In 1997 at the end of a workshop dedicated to Victor Drits by the French CNRS Bernhard Kübler proposed that it would be a great help to follow the talks if the speakers would give their definitions of vermiculite, high-charged-smectite or illite at the beginning of their talks. As a matter of fact a look through literature reveals that most of these terms are operationally defined. The aim of our study was to investigate the fate of smectite during a possible smectite to illite transformation in bentonites. The question is then, is smectite just a passive partner in the reaction giving the ingredients for the new phase illite or is illite perhaps smectite with a higher charge? In the literature two ways are described to make illite from a bentonite smectite. One is to use KCl and high temperatures (>200°C) and pressures and the other is using KOH at low temperatures (<100°C). In our presentation we will focus on the experiments with KOH.

In the study SAz-1 smectite (source clay repository) is used with 1 M KOH at 80°C to look in detail at the evolution of the smectite. XRD measurements at 40% relative humidity revealed some changes in expandability of the smectite. The 001 reflection profile of smectite was modelled using the trial-and-error approach of Sakharov (1999). The results indicate that with increasing run time the number of non expandable layers with zero or one water layer increases and that the coherent scattering domain size of the smectite decreases. Infra-red spectroscopy of the reacted smectite suggests that there is no change from the initial clay. The dehydroxilation temperature showed a slight decrease from 619° to 605°C. STA measurements demonstrated that the cis-vacant character of the octahedral sheet remained nearly unchanged throughout the experiment. The layer charge showed a continuous increase from 0.3 to 0.42 eq/Si (Si/Al)₄O₁₀. The average layer charge distribution indicated the appearance of high charged smectite layers with a charge of ~ 0.6 eq/Si (Si/Al)₄O₁₀ and the disappearance of the low charged layers. Above ~ 0.6 eq/Si (Si/Al)₄O₁₀ the smectite collapsed irreversibly.

The reaction using bentonites did not proceed through mixed-layer illite/smectite intermediates. In our experiments illite behaviour originated from a smectite via an increase of the layer charge and the final closure of the structure at values above ~ 0.6 eq/Si (Si/Al)₄O₁₀. A similar evolution can be found for smectites in KCl solutions (< 300°C). Recently Claret et al. (2004) showed that in sedimentary basins (Paris basin, Gulf coast) the smectite illitization can be described without illite/smectite intermediates being a combination of smectitic and illitic minerals of different proportions.

Sakharov, B. A., Lindgreen, H., Salyn, A. and Drits, V. A. (1999) Determination of illite-smectite structures using multispecimen XRD profile fitting. *Clays Clay Miner.*, 47, 555-566.

Claret, F., Sakharov, B.A., Drits V.A., Velde B., Meunier A., Griffault L., Lanson, B. (2004) Clay minerals in the Meuse - Haute Marne underground laboratory (France): Possible influence of organic matter on clay mineral evolution. *Clays Clay Miner.* (accepted).

THE ROLE OF ILLITE AND I-S NEOMINERALIZATION IN FAULT ZONES

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Clay neomineralization and fabric development greatly influence the behavior of faults through directly weakening caused by the low strength of clay minerals as well as through modification of the permeability structure of fault zones. Moreover, the ability to quantify fault-related changes in illite polytypism and changes in the abundance of discrete illite and I-S provide the means to directly date fault rocks through the use of Illite Age Analysis (IAA).

Illite polytypism is quantified using WILDFIRE whereas discrete illite and I-S are quantified using NEWMOD. Transmission electron microscopy (TEM) analyses of samples complement powder X-ray diffraction analyses. As with earlier studies, we observe only the $2M_1$ and $1M_d$ illite polytypes using TEM selected area electron diffraction (SAED). Therefore, in contrast to previous studies of polytype quantification which are based on mixtures of $2M_1$, $1M$ and $1M_d$ polytypes we mix $2M_1$ with $1M_d$ with varying probability of a zero rotation (a probability of 1 defines classic $1M$ illite). This allows us to account for diffuse spots observed on SAED patterns from $1M_d$ illite in our samples, which indicate that stacking is not turbostratic, but instead has limited periodicity.

Along the Moab Fault, a normal fault in southern Utah with a maximum throw of ~960 m, the most intensely deformed fault rocks are enriched in $1M_d$ illite while less intensely deformed rocks adjacent to these are enriched in I-S. As the permeability of I-S is less than that of discrete illite, the I-S neomineralization acted as a seal that confined fluids to the active portion of the fault zone, thereby providing a mechanism by which elevated fluid pressure would have weakened this fault. IAA of $1M_d$ illite-rich gouge samples and less intensely deformed I-S rich samples yield ages of ~60Ma, which represents the youngest period of activity along this fault preserved in the sedimentological record.

IAA ages from the Late Mesozoic/Early Tertiary Sevier thrust belt in western North America often yield ages of ~50 Ma regardless of their position in the hinterland-foreland progression or their location along strike. The record of synorogenic sedimentation confirms that this was the youngest period of activity along these faults, which indicates that older faults continue to play a role in orogenic evolution despite the development of younger faults.

AN INTERPRETATION OF OCTAHEDRAL ORDERING AND SITE OCCUPANCIES OF Fe^{2+} IN MUSCOVITE AND ILLITE.

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Recent experimental and theoretical, structural models have been used to infer the site occupancies of Fe^{2+} in muscovite and illite and to account for their Mössbauer spectra. Octahedral site preferences have been discussed for Al, Fe^{3+} and Mg by several authors (e.g., Drits et al., 1997; Cuadros et al., 1999; Sainz-Diaz et al., 2003; Palin et al., 2004) but, to date, Fe^{2+} has not been considered. The model proposed for the dioctahedral sheet in glauconite by Dainyak et al. (1992) that recognizes illite-like and celadonite-like domains appears to be valid for phengitic muscovites and illites as well. The trans-vacant dioctahedral sheets are separated into exclusively Al-bearing and mixed-cation domains. The mixed-cation domains have symmetrically non-equivalent cis sites with different R^{2+} and R^{3+} preferences. A high ordering energy is predicted when R^{2+} fully occupies one of the two cis sublattices in the mixed-cation domains.

Inasmuch as muscovites and illites are essentially trans-vacant, octahedral cations occupy the two cis (M2) sites in mixed-cation domains. Each cis site has three nearest neighbors on the other cis sublattice. In the lowest energy configuration, Fe^{3+} forms clusters on the R^{3+} sublattice. In accord with previous studies, it is assumed that Mg^{2+} and Fe^{2+} are randomly distributed on the R^{2+} sublattice. With this ordering, the proportions of the different local environments of Fe^{2+} cations will be determined by the Fe^{3+} -content of the dioctahedral sheet and the sizes of the mixed-cation domains and Fe^{3+} clusters. Thus, in muscovite and illite the Fe^{3+} -rich clusters within the mixed cation domains encompass much less Mg^{2+} (and Fe^{2+}) than do the Fe^{3+} -poor regions.

Mössbauer spectra of most muscovites and illites exhibit two Fe^{2+} doublets having quadrupole splittings of ~ 3.0 mm/s and ~ 2.1 mm/s. The former, which has the greater site fraction and narrower linewidth appears to represent a unique local configuration of Fe^{2+} surrounded by three Al nearest neighbors while the latter, whose splitting is smaller and more variable, appears to represent local environments of Fe^{2+} cations surrounded by from one to three Fe^{3+} nearest neighbors. Quadrupole splittings of Fe^{2+} cations will vary depending on the positions of Fe^{3+} cations relative to the crystal electric field gradient. Intermediate splittings observed in some muscovites and illites may result from cation disorder between the R^{3+} -rich and R^{2+} -rich sublattices.

Cuadros, J. et al. (1999) *Am. J. Sci.*, 299, 289-308. Dainyak, L.G., Drits, V.A., and Heifits, L.M. (1992) *Clays Clay Min.*, 40, 470-479. Drits, V.A. et al. (1997) *Clay Min.*, 32, 153-197. Palin, E.J., Dove, M.T., Hernandez-Laguna, A. (2004) *Amer. Min.* 89, 164-179. Sainz-Diaz, C.I. et al. (2003) *Amer. Min.*, 88, 1033-1045.

(Powerpoint presentation: <http://defects.physics.wsu.edu/papers/CMS-04-Illite-Symp.pdf>)

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3:20PM

ILLITIZATION AS A CRYSTAL GROWTH PROCESS

Dennis D. Eberl

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Studies of illitization in basins such as the Gulf of Mexico have shown that smectite-rich mixed-layer clays react progressively with depth to form illite-rich mixed-layer clays. At first, illite layers are randomly interstratified with smectite layers ($R = 0$ ordered); but at about 35% smectite layers the interlayering becomes ordered ($R = 1$, or IS ordered). This pattern of reaction is readily explained by nucleation of 2 nm thick elementary illite crystals while at the same time 1 nm thick smectite crystals dissolve. After smectite particle dissolution is complete, $R = 1$ ordering naturally appears at about 35 % smectite layers due to interparticle diffraction effects between adjacent elementary illite crystals that diffract coherently as stacks of two to three crystals. Thereafter, illitization consists of illite crystal growth, with or without simultaneous nucleation of new illite crystals.

To describe the growth of illite crystals, we (Eberl, Drits and Srodon, 1998, *AJS*, 298, 499-533) were led to formulate a new theory for crystal growth, because existing theories could not explain the available data. This theory starts with the observation that most minerals, including illite, commonly have lognormally shaped crystal size distributions (CSDs). The only simple mathematical method to generate such shapes by growth is according to the Law of Proportionate Effect (LPE): $X_{j+1} = X_j + \epsilon_j X_j$, where X_j is the crystal thickness after j calculation cycles (or j increments of time), and ϵ is a random number that varies between 0 and 1. If this law is applied for several calculation cycles to many crystals, a lognormal CSD results. If nucleation is continuous with LPE growth, then an asymptotic CSD is formed. These are the two most common CSD shapes for illite crystals, with the asymptotic shape forming under conditions that favor higher levels of supersaturation, such as those found within wall rock in hydrothermal deposits, whereas the lognormal CSD forms within veins in such deposits. An α - β^2 diagram (where α is the mean of the natural logs of the crystal diameters, and β^2 is their variance) and the Galoper computer program can be used to simulate CSDs and reaction paths for illite and other minerals.

Use of the LPE to describe crystal growth has several interesting consequences: (1) crystals generally will grow at a rate in proportion to their diameter, even though such growth involves adding much more volume to larger crystals than to smaller crystals during the same time interval; (2) crystal growth has a random component (ϵ), which indicates that one can not predict accurately the growth rate for individual crystals, but only for the distribution of crystals- this inherent randomness also accounts for crystal growth dispersion; (3) the relative shape of a CSD generally is determined soon after nucleation, and then is maintained during proportionate growth; and (4) illite crystal growth appears to occur discontinuously (in jumps), rather than continuously with time.

ILLITE POLYTYPES AND DIAGENESIS SINCE HOWER et al. 1976

Georg Grathoff^{*1}; Igor M. Gorokhov², and Agnes Impicini³

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Hower et al. published their seminal paper on illite diagenesis of the Gulf coast sediments in 1976. Earlier Hower authored and coauthored 2 papers in 1963 on illite polytypes and K-Ar ages of different size fractions with the observation that different illite polytypes are of different origin. Here we specifically look at how illite polytypes have enhanced our understanding of illite diagenesis.

Since Hower et al. (1976) illite polytype analysis improved significantly due to improvements in analytical and sample preparation techniques and by the help of computer modelling of X-ray diffraction patterns using e.g. WILDFIRE[©]. Illite polytype analyses help decipher the age, origin, physical and chemical conditions of illitization, by quantifying the different illite polytypes and by characterizing the individual illite polytypes. The analysis includes a) the determination of the type of polytype, $2M_1$, $1M$ cis-vacant, $1M$ trans-vacant and $1M_d$ illite and b) the determination of the disorder in the $1M_d$ illite consisting of %smectite, 60° and 120° rotations, cv/tv octahedral occupancy. In addition to a literature review we will present case studies of our own research that include: 1) Illite Age Analyses using Illite polytypes: Rb/Sr age dating of Upper Proterozoic shale of the Stangenes Formation (Varanger Peninsula, northern Norway) with a detrital age of 1.3 to 1.4 Ga and a diagenetic age of 365 to 376 Ma. 2) Physical and chemical conditions of illitization of the Campana Mahuida porphyry copper ore deposit in the Neuquén province of Argentina, where we were able to identify a high temperature illitization event forming $2M_1$ illite, formed during the evolution of the ore deposits.

References:

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- Velde, B. and Hower, J. (1963) Petrological significance of illite polymorphism in Paleozoic sedimentary rocks. Am. Miner. 48, no 11-12: 1239-1254.

STABLE ISOTOPIC EVIDENCE FOR METASTABLE ILLITE GROWTH IN A MATRIX OF NON-EQUILIBRIUM SMECTITE

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There can be significant differences in the isotopic composition of clay minerals when separated into different size fractions. This has very important implications for the interpretation of bulk isotopic data. By studying the reaction path and crystal growth patterns of illite/smectite (I/S) we might use the isotopic range of the different size fractions to infer the geologic evolution of a sample. The smallest illite crystals represent the earliest, nucleated in equilibrium with paleofluids. During burial, larger illite crystals grow and may incorporate elements from later, hotter fluids. Because larger crystals grow faster than smaller crystals (Law of Proportionate Effect), the volume of the later growth overwhelms the isotopic record of the first nucleated crystal.

A hydrothermal experiment was conducted to examine stable isotopic variations during illitization of smectite. Standard SWy-1 (K-sat) was reacted to illite at 300°C, 100 MPa in a series of sealed Au-capsules that were sequentially quenched in order to examine mineralogical and isotopic changes during reaction progress. Each capsule contained a 1:1 wt. ratio of <0.2µm SWy-1 to water containing 1000 ppm each of isotopically labeled Li and B. Boron substitutes for Si in the tetrahedral layers of illite, and Li enters the octahedral sites, therefore by examining the isotopic changes associated with crystal growth, we can evaluate the timing of cation exchange associated with different crystallographic sites. The fluid isotopic composition was changed after 30 days of reaction in order to test the hypothesis that small crystals record the earlier fluid composition.

Within the run products sampled after 30 days, we observed a 4-5‰ decrease in $\delta^7\text{Li}$ and $\delta^{11}\text{B}$ as the size fraction increased from <0.2µm to the largest crystals grown (>2.0 µm). The stable isotopic values of the smaller crystals may represent metastable equilibrium during R1 reaction progress. However, during R3 ordering, after the fluid composition was changed, the amount of illite crystallized with the new fluid far outweighs the earlier growth, resulting in no discernable difference in the isotopic composition of the different size fractions.

The isotopic composition of the mixed-layered I/S reflects illite fundamental particles growing in metastable equilibrium with the fluid, in a matrix of smectite that is out of equilibrium with the fluid and therefore masks the equilibrium isotopic record. By separating illite fundamental particles from the matrix of smectite, we might use the isotopic information of different size fractions (representing different periods of growth) to deduce fluid and/or temperature changes over time within a single sample.

SMECTITE ILLITIZATION IN ORGANIC RICH SHALE OFFSHORE MID-NORWAY

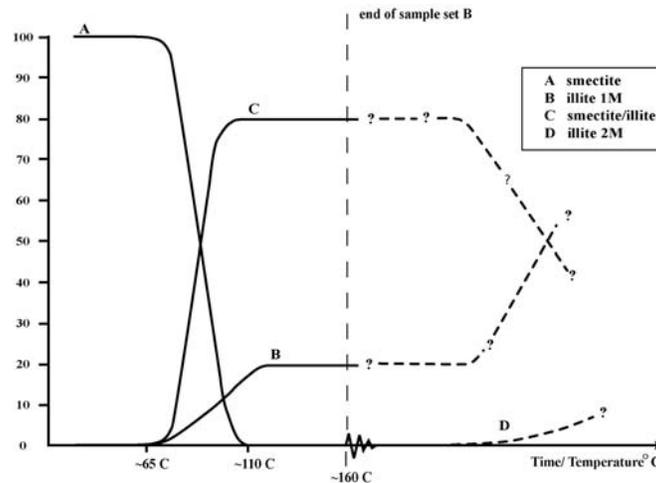
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The Spekk and Melke Formations transgressive mudstones found in the Haltenbanken area offshore Mid-Norway were deposited during the Upper Jurassic. These sediments have been subjected to continuous subsidence since deposition and constitute an excellent natural laboratory for studying diagenetic reactions.

X-ray diffraction analysis was carried out on 37 samples from 19 different wells. The mixed layer clay mineral components were identified before they were quantified using the software tool CLAY++ based on pattern fitting by a least-squares fit between the experimental XRD pattern and a suite of selected synthetic patterns generated in Mulcalc. The results from the curve fitting procedure indicate that the smectite to illite transformation occurs in distinct phases where pure smectite produces discrete illite, mixed layer illite/smectite (ML), quartz and possibly chlorite before the ML component produces illite, quartz and possibly more chlorite. The presence of detrital quartz makes it difficult to keep control on the authigenic quartz content. Anyhow, a slight increase in the quartz content is associated with the dissolution of smectite. Precipitation of quartz changes the local geochemical conditions, resulting in destabilization and dissolution of smectite and precipitation of mixed layer illite-smectite (I/S) phases, discrete illite, quartz and chlorite. There is no record of an increasing illite content in the I/S phase.

Based on this study the overall mineral reactions for the smectite to illite transformation can be summarized as follows:



Cartoon of the change in relative abundance of the various smectitic and illitic mineral phases in the sample set. The dashed lines illustrate a postulation of further development of the smectitic and illitic material below 160 °C. The plateaus exemplify how the mineral assemblage seems to stay stable between the intervals where diagenetic processes take place.