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Extracting K-Ar ages from shales: the analytical evidence

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ABSTRACT: The relationship between the K-Ar dates of mixed natural clays and the ratios of detrital and diagenetic components follows in most cases a linear trend, which agrees with the theory when the K_2O contents of the end-members are similar. However, significant deviations from a linear trend may occur and take the form of poorly-defined concave curves. The absence of a linear trend is less likely to be due to the imprecise determination of the ratio between the two end-members than to heterogeneities of the natural samples which may arise from complex K-Ar evolutions of the detrital and diagenetic end-members. The straight-line approach is of limited use in the definition of diagenetic ages in shales by extrapolating linear trends to diagenetic end-members.

KEYWORDS: K-Ar dating, illitization, shales.

The presence of detrital K-bearing minerals has often frustrated the determination of the time of illitization of detrital smectite or illite-smectite clays by the conventional K-Ar method, especially in shales and to a much lesser extent in sandstones and bentonites. In fact, biased data are certainly one reason for our limited understanding of the behaviour of clay isotopic systematics during the illitization process of detrital clay material in the limited chemical system imposed by relatively impermeable shales (e.g. review in Clauer & Chaudhuri, 1995). The whole-rock sample preparation, especially the way in which the samples are crushed and ground, may also significantly influence the data (Clauer *et al.*, 1992). This technical aspect should be of importance to investigators concerned with ascertaining the most reliable isotopic ages of clay-type materials from any sedimentary whole-rock lithology.

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As generic clay separation often leads to clay fractions with varied amounts of detrital components in them, especially in shales, some investigators have explored ways to extract the age(s) of the detrital component(s) from the measured age of a given fraction so that the true age of the diagenetic component can be established confidently. All current approaches rely on accurate determination of the proportions of detrital to diagenetic K-bearing minerals in different clay fractions of a sample and the K-Ar ages of the mixed fractions. Even so, the task of determining the ratio between detrital and diagenetic components is not easy. By assuming that these mixtures consist of two end-member components with detrital K-bearing minerals being one end-member and the diagenetic K-mineral phase the other, investigators have then mathematically extrapolated the age of the diagenetic component from the variations in the K-Ar age with respect to the proportions of the detrital and diagenetic minerals.

To the best of our knowledge, Hunziker *et al.* (1986) were the first to extrapolate the isotopic data to a pure authigenic fraction by comparing apparent K-Ar and Rb-Sr ages of clay fractions as a function of illite 1*M*/2*M* ratios. Liewig *et al.* (1987), after quantifying by X-ray diffraction (XRD) the amount of detrital feldspar in mixed diagenetic illite-detrital feldspar samples, explored the use of a correction factor derived from quantitative estimation of the rate of change in the K-Ar age as a function of the detrital component. Hamilton *et al.* (1989) used a similar approach to determine the effect of detrital contamination on the age of diagenetic clays. Ehrenberg & Nadeau (1989) and Mossmann (1991) pursued a similar course by establishing the amount of the detrital component in a given clay separate by optical, X-ray, or chemical means that allowed correction factors to be applied to the measured ages of mixed-clay samples. Later, Pevear (1999) suggested almost the same approach, but with more specific means of determining the authigenic/detrital illite ratio for removing the overprint of the detrital age on the age of a mixed diagenetic-detrital clay sample. As estimates of the proportions of diagenetic illite mineral in detrital-authigenic mixtures need to be very accurate for precise determinations of K-Ar ages, Pevear (1999) recommended two approaches of quantification that rely on XRD analysis. Like Mossmann's (1991) use of a decomposition mode, Pevear (1999) proposed a decomposition mode based on the NEWMOD technique of Reynolds (1985), as well as a calculation of the illite polytype percent, with the assumption that the detrital and the authigenic minerals are of different polytypes, as Hunziker *et al.* (1986) did, assigning the 2*M* polytype to the detrital phase and the 1*M*/1*Md* polytypes to the authigenic phase. Similar fundamental steps in this approach were advocated earlier by Clauer & Chaudhuri (1995). Pevear (1999) is correct to point out that the new methods for quantification are crucial to the derivation of the age of illite diagenesis, but it is also essential to keep in mind that the stated extrapolation works well only when analysed samples meet two other important criteria: (1) the samples must contain no more than a single-age detrital component; and (2) the diagenetic illite must have formed essentially during a single (short) episode.

Recently Środoń (1999) envisaged a purely theoretical approach to assess the mixing effect on K-Ar data for illite mixtures, by defining a series of computer-generated concave downward, concave upward and straight line trends for the K-Ar data by mixing varied proportions of a diagenetic illite and a detrital illite. He showed that the trend of the mixed age is concave downward when the detrital illite has a higher K_2O content than the diagenetic illite, concave upward when the detrital illite has a lower K_2O content than the diagenetic illite, and linear when the two illites yield similar K_2O contents (see his Fig. 1). To evaluate what Środoń's (1999) theoretical modelling may bring to the debate on extraction of reliable K-Ar ages of authigenic illite formed in shales, we believe that it should be compared to analytical data obtained from natural samples. The analytical reality is not based on pure homogeneous detrital/authigenic illite mixtures, but on heterogeneous end-members often mixed with minor mineral phases that may contain varied amounts of K_2O, such as micas and K-feldspars. Also, secondary illite often results in shales from illitization of illite-type precursors and not from illitization of smectite, and the effects of such an illitization process on the isotopic systematics of the precursor progeny are not yet clearly understood.

In the current resurgence of efforts to define diagenetic ages by extricating the influence of the detrital end-member age from measured ages, we feel that a revisitation of mixed-age patterns that have emerged from studies of natural samples would be instructive. The results from analyses of natural clay samples could document whether or not a linear mixed-age pattern is normal or if a mixed-age pattern has concave affinities. A major focus of this paper is to document the shapes of mixed-age patterns of natural samples and provide some rational explanations for them. Any clear departure from a linear trend for mixed-age clay fractions, assuming no large errors in the estimates of component abundances in a two-component system, signifies that the different size-fractions cannot be defined just by two end-members with distinctly different ages. The non-linearity then indicates that the bulk clay material consists of a system with more than two end-members.

THEORETICAL AGE PATTERNS FROM MIXTURES OF DIAGENETIC AND DETRITAL CLAYS

Almost the entire amount of K and Ar is hosted by three types of minerals in an average shale, namely detrital K-feldspar, detrital mica or illite, and diagenetic illite (including mixed-layered illite-smectite). In some special circumstances, such as in bentonite beds, the K may be hosted by glassy material. Hence, the relative proportions of these minerals and the relative differences in their ages define various K-Ar mixed-age patterns. To limit our discussions to relatively simple detrital histories, we have chosen to focus on illite as being the primary detrital mineral phase. We are fully aware that the presence of detrital K-bearing feldspars could have significant influence on the character of mixed-age patterns. This influence may be small in dealing with clay-size particles because feldspars are found to occur commonly in larger grain sizes. Four different diagrammatic representations of mixed-age patterns (Fig. 1) are given, corresponding to varied mixings of different illitic components. We do not consider these patterns to be inclusive of all mixed ages. The contexts of the mixed-age patterns in our discussions include mixings: (1) between an homogeneous K-bearing detrital illite of a given age and an homogeneous authigenic illite mineral with a younger fixed age, the two components differing in or having similar K_2O contents (Case I); (2) between heterogeneous detrital illites with varied ages that were inherited at the time of deposition, and an homogeneous authigenic illite with a fixed age (Case II); (3) between heterogeneous detrital illites with varied ages that were inherited during burial, possibly in connection with illitization, and an authigenic illite with a fixed age (Case III); and (4) between an homogeneous detrital illite with a fixed age and a heterogeneous authigenic illite with varied ages (Case IV).

Case I – Mixing in different proportions between a detrital illite of a fixed age and an authigenic illite of a younger fixed age

Mixing between an homogeneous detrital component with a specific age and an homogeneous authigenic mineral with a known age will define one of the three trends calculated by Środoń (1999), who considered the effects of relative K_2O contents and fractional abundances of the components (Fig. 1, Case I). Such mixed-age trends are most likely to occur only in bentonite deposits, in contrast to that from shale and sandstone units where heterogeneous detrital end-members are common. In spite of some apparent success in deciphering the period of illitization in shales by extrapolation of mixed-age linear trends to the zero per cent detrital component (Pevear, 1999), we contend that great uncertainty exists regarding the fixing of time of illitization in shales by such a simple method based on data for three size-fractions separated from a sample of an average shale, because of the age heterogeneity that is inherent in any detrital material. Major concerns in dealing with average shales are illustrated in the next three diagrammatic representations of potential mixed-age patterns.

Case II – Mixing between a detrital component containing two phases that differ in their inherited ages (which were not affected after deposition) and a younger authigenic phase

Multicyclic sediments are found in abundance in the sedimentary record. Hence, in the simplest case of two detrital illitic or micaceous components in association with an authigenic illite in a sample of shale, the different separated fractions will consist of a three-component system with varied abundances of the components. This will result in varied mixed ages with no great probability that any definable linear trend may be observed (Fig. 1, Case II). Any distribution of data points within the area defined is possible, resulting from varied mixings of the components. An accurate extraction of an authigenic illite age requires a good knowledge of the ages of the two detrital components.

Case III – Mixing between a detrital component the K-Ar age for which evolved during burial and an authigenic component that formed during a single episode

Shale beds may contain detrital minerals that could have been derived from a single source or several sources with a single age or a very narrow age span. A possible environment for such a case could be a simple geological setting without tectonic activity in which a young sedimentary sequence essentially derived from a small drainage basin, was buried rapidly. Another example could be a molasse deposit with detrital minerals derived from nearby source rocks with a limited range of ages, but may have been disturbed by some post-depositional alteration. Basically, any detrital component is variably altered during the post-depositional burial history of its host rock, resulting in selective losses of ^{40}Ar with consequent varied

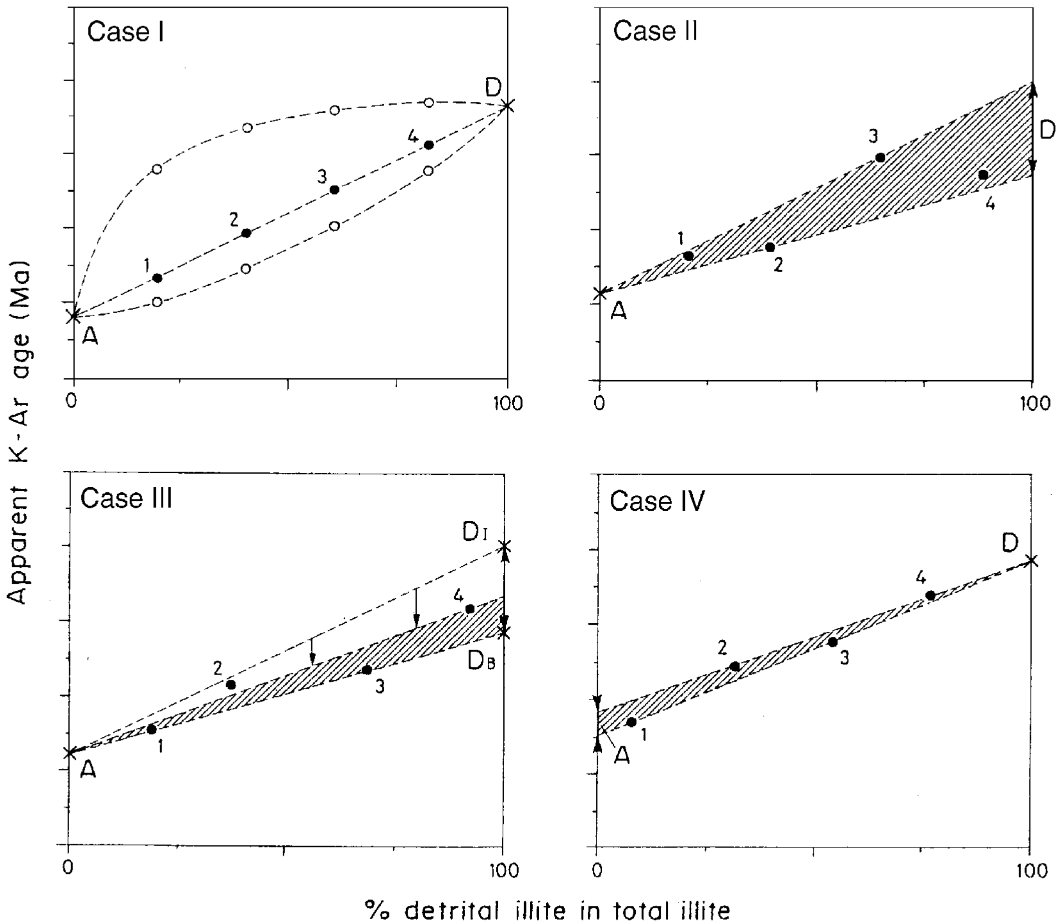

FIG. 1. 1 Patterns of mixed K-Ar ages from mixtures of detrital (D) and authigenic (A) K-bearing minerals (illites in the present context). Case I outlines the patterns of mixing between a detrital illite with a fixed age and an authigenic illite with a younger fixed age, depending on the K_2O contents of both (Środoń, 1999). Case II outlines the pattern of mixing between a young authigenic phase and an older detrital component consisting of two phases that differed in their inherited ages at the time of sedimentation, these ages not being affected after deposition. Theoretically the data points should occur in the shaded area. Case III shows the pattern resulting from mixing a detrital component, the K-Ar age of which evolved during burial, and an authigenic component formed during a single episode. D_I stands for initial detrital and D_B for buried detrital. If altered by burial, the fractions are scattered within the dashed area. Case IV outlines the pattern resulting from mixing a detrital component with a fixed age and authigenic components that differed narrowly in their ages. The dashed lines represent the limits of the surface in which the data points plot. The numbers outline four theoretical size fractions of a sample from finer (1) to coarser (4).

reductions in ages. Such selective losses were studied by Hunziker *et al.* (1986) on detrital illite of slightly metamorphosed shales. Such alteration effects which may have been induced mainly by temperature increase essentially on single species of K-bearing detrital minerals, are unlikely to produce a linear trend for the mixed-age patterns in proportion to the fractional abundance of the authigenic component. A more likely situation would be that the data points are scattered in an area limited by the highest and lowest ages of the detrital end-members and the age of the authigenic illite (Fig. 1, Case III). This actually appears to be another way to produce a multiple age relationship already envisaged in Case II. The following discussion that relates to a specific case study will reveal a significant difference between the two cases.

Case IV – Mixing between a detrital component and authigenic components of differing ages having a limited range

A few isotopic studies of old sedimentary basins with long evolutionary histories have shown that sedimentary rocks having multi-episodal secondary illites are more common than those having mono-episodal secondary illites (e.g. Lee *et al.*, 1989; Mossmann *et al.*, 1992; Gorokhov *et al.*, 1994). In theory, the mixed-age patterns from clays with multi-episodal illites will be the same as if they were from two detrital components (one of which is the early diagenetic component) and an authigenic component (in this case the latest diagenetic component). If the two diagenetic components differ in their ages by a small but measurable amount, the data points for the size-fractions would be scattered in a restricted area and a simple linear trend certainly might exist (Fig. 1, Case IV). Of course, authigenic illite of two generations only coexist if the younger one was formed at lower temperature than the previous one (see discussion in Clauer & Chaudhuri, 1995). The task of identifying the presence of two illitic components may not be simple by means of XRD data.

NATURAL AGE PATTERNS FROM MIXTURES OF DIAGENETIC AND DETRITAL CLAYS

From the theoretical approach above, it appears that in most case studies, the mixed-age patterns will not be strictly linear but the data points will probably scatter around arrays that are anchored at the age(s) of the authigenic illite. Non-linearity, theoretically, can be limitless, from concave to barely pseudo-concave in nature, due to continued or episodic K-Ar gain/loss by either of the two components or both, or to a situation wherein a diagenetic component incorporated an excess of radiogenic Ar during crystallization. Because of these factors, which are common in shales, the clay system becomes in essence a multicomponent system with more than two end-members, suggesting that non-linear trends very often cannot be independently dissected mathematically, or that mixed values may not be extrapolated, to define with a high degree of confidence the ages of the end-members. This limitation is particularly critical for the age of the diagenetic end-member which is often the major goal of K-Ar analyses of clay minerals.

The discussion below is based on a study by Furlan (1994) who presented a detailed mineralogical, geochemical and K-Ar study of illitization in buried shales and associated sandstones of the Mahakam delta, with respect to progressive burial depth. The data represent an informative analytical basis to evaluate the straight-line theory of Pevear (1999) and the theoretical test of Środoń (1999) to extract K-Ar ages from shale fractions that contain various illite-type materials, because not only do they provide mixtures of authigenic and detrital end-members, but also their K-Ar values are based on independent determinations. Furlan's study (1994) of the Mahakam delta provided XRD and K-Ar results for several size-fractions (<0.2, <0.4, 0.2–0.6, 2–6, 2–40, 20–40 and 40–63 μm) that were extracted from three shales buried to 183, 1000 and 4232 m, respectively, and from associated sandstones of the same borehole at 1878 and 4228 m. As in previous studies, NEWMOD decomposition (Reynolds, 1985) of the XRD data allowed calculation of the detrital illite component in the mixtures of the shales and the sandstones. The Mahakam delta is very suited to the present discussion, because: (1) the Mahakam River drained a small basin limited by mountains whose geological history is well documented; (2) the isotopic age of the detrital material is known independently; (3) the stratigraphic age of the two deepest rocks is fairly well known; and (4) the burial-related progressive evolution of the same minerals can be followed in shales and sandstones during progressive burial, in order to evaluate the influence of burial alteration on the K-Ar systematics of the detrital end-member. An integrated mineralogical, geochemical and isotopic study of the illite-type minerals of the shales and the sandstones in one oil field of the Mahakam delta, also showed that the evolution of the K-Ar system in the clay fraction of the shales is different from that of the sandstones (Clauer *et al.*, 1999).

The stratigraphic age of the deepest units reached by drilling at ~4000 m is known to be at ~18–20 Ma. Also, the diagenetic effects were much less pronounced in the shale-associated illite, yielding a K-Ar age of 50 Ma at a depth of 4000 m, than in the sandstone-associated illite, providing a K-Ar age of 20 Ma for the <0.2 μm fraction (Clauer *et al.*, 1999), and of ~15 Ma for the separated fundamental particles of <0.02 μm size (Clauer, unpubl. data) at nearly the same depth. In the uppermost shale sample buried to 183 m, the K-Ar age of the detrital illite can be considered to be ~120−140 Ma, whereas the authigenic illite should theoretically yield 0 Ma. The known K-Ar ages of the detrital and authigenic illites allow either the theoretical straight line or the triangular area representative of the mixtures of the components to be drawn. This information, shown as an area in Fig. 2, is basically unknown when applying the straight-line theory to extract the K-Ar age of an authigenic shale-derived illite. The K-Ar data points of the uppermost shale buried to 183 m plot in the upper right side of the diagram (Fig. 2a) with amounts for the detrital component ranging from 50 to 95% in the different size-fractions. They show clearly that extrapolation of a K-Ar age for the authigenic illite component in such a mixture is not straightforward: the data points define a quadrangular area, even wider than the area defined by the known K-Ar values. The best-fitted line through the data points provides a detrital K-Ar value of ~120 Ma which is reasonable, and an authigenic K-Ar value of ~10 Ma, which is significantly above the expected 0 Ma age. It might be added that the occurrence of ~50% authigenic illite in a mixed-layer form at this shallow depth probably corresponds to a mineral which crystallized during a pedogenic episode before being transported to the delta of the river. This intermediate pedogenic episode may partly explain the large scatter found in the mixed-age pattern.

The shale sample buried to intermediate depth at ~1000 m, contains mixtures in the separated fractions showing varied amounts of detrital illite, from ~30% to ~90%. The data points plot closer to a linear array than in the shallower shale. However, this array has a slightly steeper slope than the theoretical area drawn on the basis of the independent age information of 120−140 Ma for the detrital end-member and ~5 Ma for the authigenic end-member (Fig. 2b). The extrapolated value from the straight-line approach for the authigenic illite is ~15 Ma, which is again significantly above the known age estimation for the illitization process at this depth.

In the shale buried at 4232 m, the detrital illite contents vary in the different size-fractions from 5% to 95%; the shaded area based on the respective ages of the detrital and authigenic end-members is also provided (Fig. 2c). The pattern of the data points is very different from those of the two shallower shales. It is of a concave downward type marked by a change in the slope near the authigenic component. The apparent linear trend with a low slope among the three coarser fractions and an addition of an element of non-linearity to the trend by the incorporation of the data from the very fine fraction (<0.2 μm) outlines two important new aspects: (1) the apparent age of the detrital material decreased dramatically between 1000 and 4232 m from ~120−140 Ma to ~60 Ma; and (2) the extrapolation of the authigenic illite age is very different from the theoretical value, ~45 instead of 15 Ma, as it was for the shales from shallow and intermediate depths.

As the authigenic illite from sandstones appeared to be much more evolved than that from shales, probably because of a different illitization process (Clauer *et al.*, 1999), the data of the two deepest shales were compared to those of nearby sandstones. At an intermediate depth of 1878 m, the size-fractions of the sandstone sample contain ~30−90% detrital illite, which is close to the values of the shale at 1000 m. The age pattern of this sandstone is also very similar to that of the shale at 1000 m with a collective plot near a linear array that is close to the theoretical line. The lower intersect of the analytical line is at 10 Ma which is very close to the theoretical value of 8 Ma on the age determined on fundamental particles from a deeper sandstone. As in the deepest shale, the size-fractions of the deepest sandstone at 4228 m, contain between 10 and 95% detrital illite. The age pattern of this deep sandstone is similar to that of the sandstone at the intermediate burial with a linear distribution for the data points. The intercept on the authigenic side is again near 15 Ma, which is reasonable with respect to the dated fundamental particles, while the detrital age extrapolation shows a significantly lower value as did the detrital end-member of the deep shale.

A real difficulty in extracting a reasonable authigenic illite age from progressively buried shales of the Mahakam basin is in the deepest sample, where diagenesis may have been the most penetrative. The temperature estimated by fluid-inclusion microthermometry was at 180−190°C at

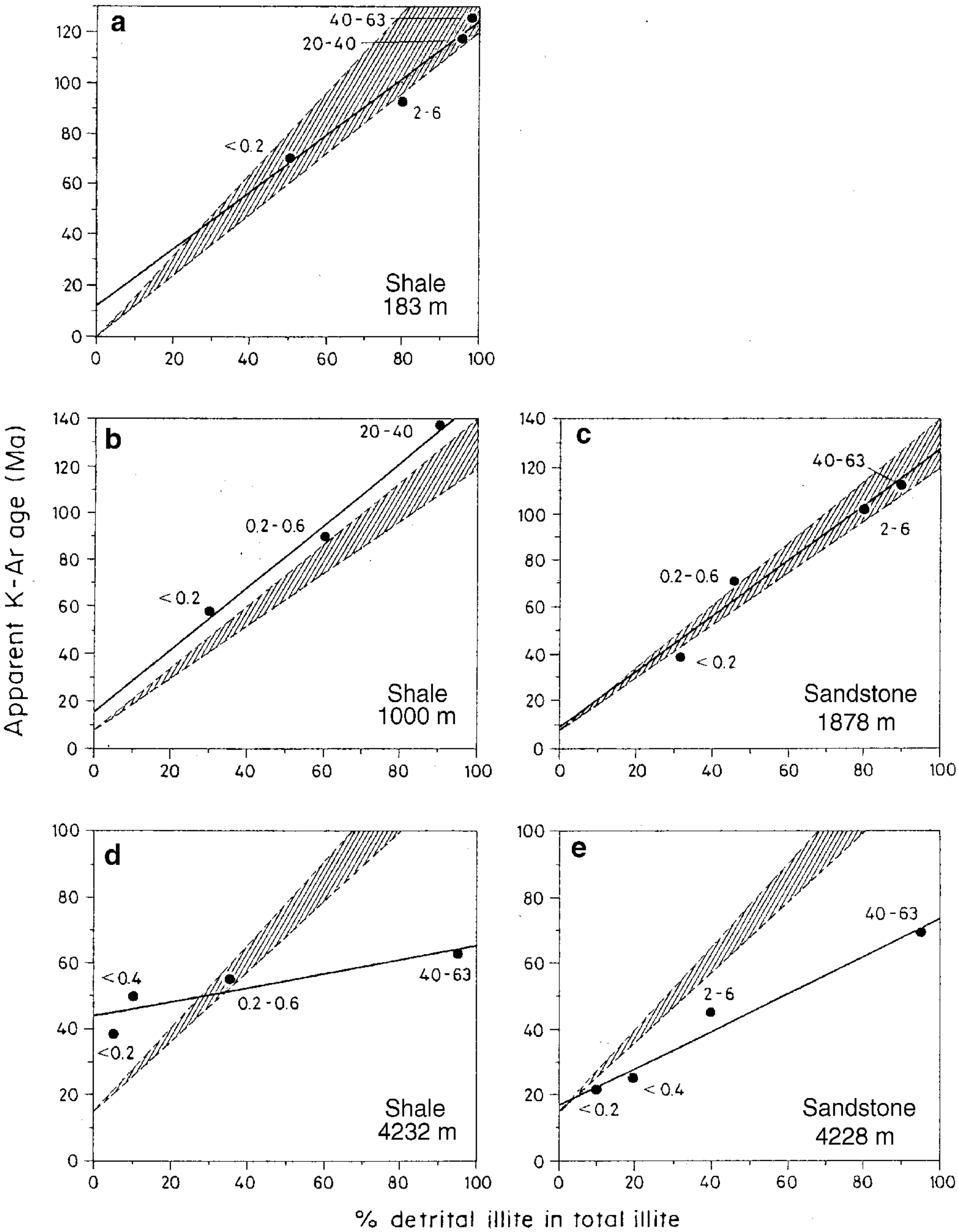

FIG. 2. Apparent K-Ar ages of several size-fractions from natural shales and sandstones (sizes given next to the data points in μm) with respect to their varied ratios of detrital and authigenic illite-type minerals. Parts a, b and d show the data for the different size-fractions from three shales buried to 183, 1000 and 4232 m in the sedimentary sequence beneath the Mahakam Delta. Parts c and e outline the data for different size-fractions from two sandstones buried to 1878 and 4228 m in the same sedimentary sequence. The lines represent the 'straight-line' extrapolations discussed in the text, whereas the shaded areas are defined by the ages of the authigenic and detrital components as discussed in the text.

~4225 m, depending on the assumed trapping pressure. The occurrence of a penetrative diagenesis is also suggested by the significant lowering of the K-Ar data of the detrital end-member in both the deepest shale and sandstone. The measured mixed-age pattern indicates that the K-Ar dates of the two fine fractions (either of <0.4 or <0.2 μm size) are anomalously high with respect to the theoretical mixed-age line that connects the age of provenance of the detrital illite in the shales to the age of illitization. The uncertainties in the estimates of the amount of detrital minerals in these finer fractions will not bring the data points closer to the theoretical line, as this analytical aspect is not influenced at all by the same relationships in the other samples. An interesting explanation for the anomalously high K-Ar data of the two finest fractions in the shale could be a gain of ^{40}Ar in the authigenic illite forming in the shale, while this gain did not occur in the nearby sandstone. This excess of ^{40}Ar could have been derived from intimately mixed detrital illite which lost amounts of radiogenic ^{40}Ar relative to K_2O, as its overall K-Ar age decreased significantly from ~130 Ma to 60 Ma. The detrital illite from associated sandstone also lost large amounts of radiogenic ^{40}Ar which probably escaped due to greater permeability. The non-linear, concave downward trend in the deepest shale could therefore be indicative of a specific illitization process and not an analytical artefact. Furlan's (1994) data from shallowly buried shales and sandstones (<1900 m) relate to Case I, whereas the deeply buried shale and sandstone (>4200 m) belong to Case III with a complication for the shale which may distract from obtaining K-Ar ages for authigenic illite from any mixture with detrital illite. If this relationship is confirmed by further studies of shales that underwent significant diagenetic alteration, extracting K-Ar ages for the authigenic illite by straight-line extrapolation is virtually impossible.

DISCUSSION: IS A LINEAR TREND A RELIABLE ASSUMPTION FOR THE MIXED AGES?

The fundamental point, as outlined by the different examples of natural size-fractions from shales and sandstones taken at progressive depths along bore holes, is that mixed-age patterns from data from different size-fractions of a sample often correspond to a simple linear trend with respect to presumed mixings of two end-members with different ages. Mixed-age linear trends with respect to the detrital-authigenic illite ratio are possible when the samples consist of two end-member components with distinctly different fixed or invariable ages. Only one non-linear trend was observed and it seems to result from the effects of a specific situation combining the intensity of the diagenesis, the rock type and its inherent physical properties. In this case, extraction of a K-Ar age for the authigenic illite is not possible. While a good linear trend depends on high-quality quantification of the fractional K abundance of an end-member in a two-component system, it results also from the inherent demand that the system be represented by two components with fixed or invariant ages. This may explain why Pevear (1999) envisaged a general applicability of the linear extrapolation of the K-Ar age-mineral abundance data to define the age of illitization in shales.

The clays in bentonites, on the other hand, are reduced to two end-members with different ages which are often fixed or only slightly varied (Clauer *et al.*, 1997). Considering that a two-member secondary mineral history is likely to be the case for many bentonite deposits, a linear extrapolation from mixed-age data for the isotope age analyses of a diagenetic end-member may work very well for such systems. But the universal applicability of the extrapolation approach is highly questionable, especially in the case of shales that were exposed to strong diagenetic conditions, possibly not allowing radiogenic ^{40}Ar released from altered detrital illite to escape from the immediate environment of the authigenic illite.

CONCLUSION

Theoretically, apparent K-Ar dates of different clay fractions separated from a shale (or from a sandstone) containing a clay of detrital origin and a clay of diagenetic origin, should depend linearly on their K_2O contents, which are often quite similar for the two components, and to their proportions in the separated fractions. But in some circumstances, as reported here, the data deviate greatly from linearity and may describe poorly-defined concave curves. The absence of a linear trend, which does not allow the straight-line theory to be used universally, is less likely to be due to an imprecise determination of the ratio between the two end-members, but has more to do with the vagaries of the natural samples reflecting complex K-Ar evolutions of both the detrital and diagenetic end-members. The straight-line approach has limited applicability to define diagenetic ages in shales by extrapolation of the linear trend to the diagenetic end-member; its application to sandstones and bentonites seem to be much more promising.

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