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# RE-EXAMINATION OF SOME Rb-Sr AND K-Ar AGE DETERMINATION OF CRETACEOUS AND TERTIARY DEPOSITS IN BELGIUM AND THE ADJACENT COUNTRIES

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SAMENVATTING. - Een zeker aantal geochronologische gegevens betreffenden glauconieten uit Belgie en Noord-Frankrijk werden vroeger reeds gepubliceerd. De kennis in verband met het glauconiet liet het ons toen niet toe die gegevens van een grondige interpretatie te voorzien.

Dankzij recente werken, die dikwijls slechts een verduidelijking vormen van oudere ideëen en dankzij en vergelijking van verschillende gevallen, lijkt het nu mogelijk de bijkomende informatie te verschaffen die toelaat, in grote lijnen, het geochronologisch gedrag van het glauconiet te schetsen.

Afgezien van de eigenlijke dateringen, kunnen radiometrische bepalingen worden opgevat om bij te dragen tot het oplossen van andere problemen, zoals herwerking, verwering, diagenetische evolutie, enz..

RESUME. - Un certain nombre de données géochronologiques publiées sur glauconies de Belgique et du Nord de la France n'ont fait l'objet, jusqu'à ce jour, que d'une interprétation sommaire, faut d'éléments permettant une interprétation plus poussée. A la lumière de travaux récents, qui reprennent souvent des idées anciennes, en les précisant, et par la comparaison de différents cas, il apparaît possible de dégager le complément d'information permettant, dans les grandes lignes, de prévoir le comportement géochronologique de la glauconie. Indépendamment de la datation proprement dite, des mesures radiométriques peuvent être conçues en fonction de la solution d'autres problèmes : remaniements, évolution diagénétique, altération, etc..

SUMMARY. - A number of published geochronologic data relative to glauconite from Belgium and North France remain so far without interpretation, for lack of informations which would make such interpretation possible.

In the light of some recent work, sometimes based on ancient concepts, which are further precised, and from the comparison of different cases, it seems possible to provide these complementary informations needed. Tentatively, the "geochronologic behaviour" of glauconite can be sketched.

Besides the dating of sedimentation time, radiometric measurements may be used for the purpose of elucidating cases of reworking, diagenetic evolution, weathering, etc..

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## INTRODUCTION.

The interpretation of radiometric ages on glauconite has led in the past to considerable discussions. Together with an improved knowledge of its mineralogy, crystal chemistry, process of formation, a clearer picture of its isotope geochemistry is beginning to emerge. The purpose of this paper is to attempt to sketch this picture, and simultaneously, re-discuss some of the data published by our group (data which have been obtained in collaboration with many Belgian geologists, and, for some of the radiometric data, J. C. HUNZIKER, and G. S. ODIN).

An introductory rash statement to the difficult problem of interpreting glauconite age, read in many papers and textbooks is that "radiometric ages on glauconites are generally 10 to 20 % too low as compared to high temperature mineral data". This conclusion of the earlier contributors (e. g. HURLEY et al., 1960) insofar it is considered as a general rule, has not been confirmed by subsequent work, and may even be considered as misleading (BROOKINS, 1976; KEPPENS and PASTEELS 1981, in press). It has been shown by OBRADOVICH (1964) that glauconite K-Ar ages are often lower than that of sedimentation, but also that this lowering is variable. ODIN (1975) has attempted to define the criterions which allow the selection of glauconites suitable for K-Ar dating. Absence of burial and heating, weathering and reworking, and a content in expandable layers not exceeding 15 % seem to be, if not necessary, possibly sufficient conditions for the obtention of significant radiometric ages.

Radiometric measurements may however be performed on glauconites which do not bear such features, in order to check the validity of the selective criterions, or the possibility of deriving some informations on the sedimentary or diagenetic evolution or the glauconite genesis. In such cases, radiometric dates must be considered as "apparent ages" only.

In what follows, the different factors which may influence glauconite apparent ages will be considered in turn, illustrated from examples taken from our own work, whenever possible. A case where those different factors may combine their effects is discussed in fine.

## BURIAL AND HEATING.

This situation is mentioned for completeness. Indeed, glauconites from the Cretaceous and Tertiary from North France, South England, Belgium, collected in outcrops or drill-holes at shallow depth are free from these influences which tend to lower the K-Ar age and to a lesser extent the Rb-Sr age, as shown in many papers (reviewed by KEPPENS and PASTEELS, 1981, in press).

The fact that in the early development of glauconite dating, many samples have been collected in consolidated, or even tectonized rocks provides a partial explanation for the subsequent disgrace of glauconite as material for datation. Paleozoic or Precambrian glauconites may however yield acceptable age indications in stable areas as the Russian Platform (reviewed by AFANASIEV and ZYKOV, 1975) or some parts of the North American continent (OBRADOVICH and PETERMAN, 1968).

## GLAUCONITE FORMATION.

Glauconite formation proceeds at low temperature and, even at the geologic scale, is a slow phenomenon. The question thus arises whether there is a "zero time" at which no daughter nuclide is present in the sample and, if so, to which geologic event it relates.

Theories explaining the formation of glauconite are diverse and, as noted by Mc RAE (1972), should not be considered as mutually exclusive.

1. An iron-aluminium-silicate gel would act as glauconite precursor (Takahashi and YAGI, 1929; SEED, 1968).
2. Alteration of biotite (GALLIHER, 1935) or muscovite, chlorite, etc. This theory may account for specific cases, but its importance seems to have been overempharized (HEIN et al., 1974; ODIN, 1975).
3. Attraction of iron and potassium by a degraded 2 : 1 layer silicate latice (BURST, 1958, HOWER, 1961). A gradual uptake of Fe and K takes place, leading to an interlayering of iron-rich smectite and of the Fe polymorph of illite. In the course of time, the proportion of expandable layers decreases with increasing potassium content. The theory is generally accepted for the evolution of glauconite with a large content in expandable layers towards "mineral glauconite". But that the glauconite precursor is a degraded 2 : 1 layer silicate in all cases remains a matter of debate.
4. Replacement. Epigenesis of various minerals by glauconite has been observed, namely carbonates (CAYEUX, 1932; LAMBOY, 1968, etc.).
5. Precipitation. Though glauconite formation needs a suitable substrate to start (often, a sand-sized, porous particle) some observations would point to a limited growth of the initial pellet by precipitation (neoformation) (LAMBOY, 1975). In the case of glauconite formation in the test of Foraminifera, it is often postulated that the infilling of the test which upon alteration is converted to glauconite, is predominantly detritic clay. However it might also be a colloïd, according to EHLMANN et al. (1963) or a "crystallizing smectite-type material" (ODOM, 1976).

From this it can be concluded that glauconite is actually formed, in some cases, at the expense of minerals which carry no radiogenic strontium, such as quartz or carbonates, or by precipitation. Excess daughter nuclides is expected to be present only in immature glauconites formed at the expense of silicates. Maturation of glauconite results apparently, from the many radiometric data available, in the expulsion of this radiogenic argon or strontium in excess.

Excess argon has been observed on some glauconites with a large content in expandable layers (OWENS and SOHL, 1973; ODIN, 1978; TISSERANT and ODIN, 1979). The presence of radiogenic strontium in excess is to be expected, in the same cases as those where excess argon is observed, and for the same reasons. So far very few comparisons have been made of Rb-Sr and K-Ar ages for K-poor, immature glauconites.

From one investigation, still in progress (PASTEELS and KEPPENS, in preparation) it would appear that as much or even more inherited radiogenic strontium than argon may be present in glauconite with a high content in expandable layers. The Clay of Flanders Member of the typical Ypresian contains an intercalated glauconite-bearing silty bed. This glauconite has a 4 to 5% K content corresponding to about 20 to 30 % expandable layers. Apparent Rb-Sr ages are about 10-25 Ma higher than that of sedimentation. A more detailed mineralogical study would be necessary in this case before concluding on the probable cause of this excess strontium (or rubidium defficiency). Some analogies may however be drawn between this and other cases. From available data this glauconite is not very different from that of the Upper Ypresian of the same region, which has been investigated more in detail (though, to our knowledge, not by the means of geochronology) (ODIN et al., 1972; VELDE, 1976). This latter is considered to be partly at least derived from a silicate frame, and has been compared to the glauconites originating from fecal pellets occuring on the shelf off the coasts of Gabon and Congo (GIRESSE and ODIN, 1973).

Another probable case of excess radiogenic strontium and possible minor excess radiogenic argon, is that of sample R 23-24 (two size fractions) of the Upper Aptian at Wissant, Pas-de-Calais (ELEWAUT and ROBASZINSKI, 1977; KEPPENS et al., 1978). It has a K content of 5,2 % corresponding to about 20 % expandable layers, and occurs in a clayish and silty sand layer. Its Rb-Sr apparent age is $115\pm$ 3 Ma and K-Ar apparent age $106\pm$ 3 Ma. Other glauconites from the Cretaceous have a much lower content in expandable layers and yield concordant ages (Fig. 1). Most of these well-ordered glauconites present themselves in sediments containing little or no clay (same ref. and HUNZIKER and ODIN, 1981). At the same locality, very consistent data are obtained on mature glauconites from the Albian, pointing to a $100\pm$ 3 Ma age for the transition lower Albian-middle Albian. Due to the small number of radiometric data for the Aptian stage, the absolute age of the upper Aptian is not too well known. Nevertheless, a figure close to 106 Ma (apparent K-Ar age of sample R23-24) would appear as a reasonable extrapolation, the 115 Ma figure (apparent Rb-Sr age of the same sample) being definitely at the high side.

The internally consistent dates for the Upper Cretaceous, mentioned above, and those of PRIEM et al. (1975) are either in excellent agreement with, or else up to 5% lower than comparable dates on high temperature minerals (OBRADOVICH and COBBAN, 1974). Considering the uncertainties associated with intercontinental correlations, this small difference may be not significant. Alternatively, it may reflect the fact that the radiometric clock starts, when the maturation of glauconite ends, i.e. when it is buried under sediments (ODIN, 1975). Glauconite would thus date, not the sediment in which it occurs, but in some cases at least, the first overlying deposit. This may be significant geologically, since glauconite maturation requires a low or negative sedimentation rate and glauconite occurences, often correspond to sedimentary breaks (e. g. GOLDMAN, 1922; CLOUD, 1955; HOWER, 1961).

## REWORKING.

On the subject of glauconite reworking, opinions are conflicting and some criterious such as the morphological ones (e. g. LIGHT, 1952) are probably of little value. Glauconite pellets are probably transported over short distances as a rule, bu so are most fossils, which are nonetheless considered as useful in biostratigraphy. Rounded pellets may be formed by limited growth around a nucleus (LAMBOY, 1975) and are thus not the result of abrasion in all cases. In cases where glauconite reworking was considered a constant phenomenon, such as the Cretaceous and Tertiary greensands of New Jersey (OWENS and MINARD, 1960), radiometric data have shown that reworking was in fact rather exceptional (OWENS and SOHL, 1973).

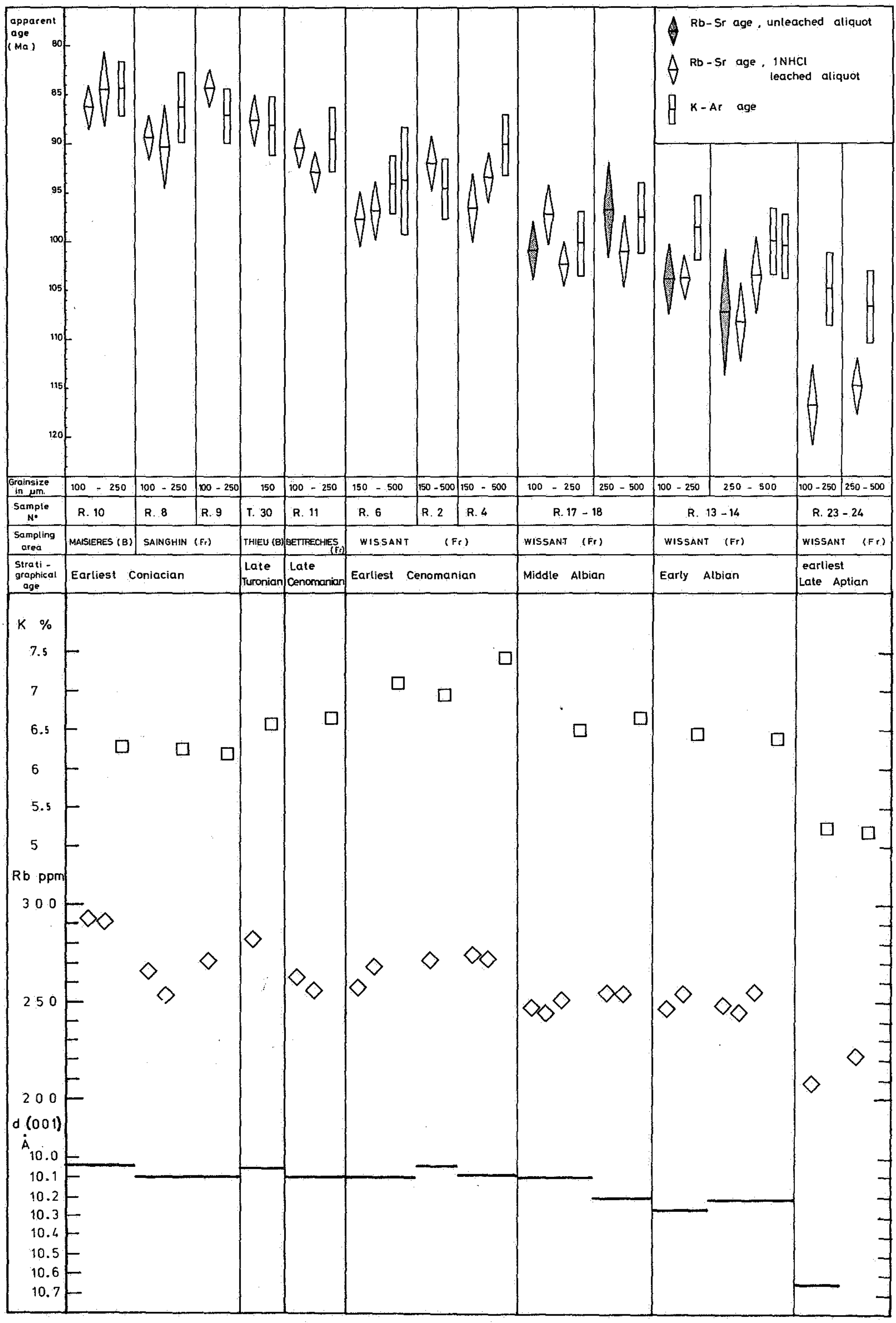



Fig. 1 - Glauconites from the Cretaceous.
Apparent ages, K and Rb contents, d(001).

WERMUND (1964) has shown that fluviodetrital glauconite should not influence radiometric ages significantly, except for sediments containing less than 2% glauconite pellets. This is due to dilution more than abrasion. Laboratory experiments (same ref.) show that glauconite pellets may be transported over long distances. The probability of glauconite being reworked is thus more or less comparable to that of fossils of the same size-class. Uncertainties still subsist on the subject of the hydrodynamic properties of glauconite compared to those of carbonates, and other minerals, such as quartz.

One case in the Belgian Neogene deserves mention. According to GULLENTOPS (1957, 1963) the glauconite of the Diest Formation (DE MEUTER and LAGA, 1976) is reworked. His arguments, based on sedimentologic and geomorphologic considerations, are fully confirmed by radiometric dates (ODIN et al., 1974a; KEPPENS, 1981). This Upper Miocene deposit contains indeed glauconites yielding apparent ages ranging from 15 to 20 Ma (Rb-Sr and K-Ar) which is about twice the probable age of sedimentation. The K contents, 6,1 to 6,2 %, correspond to mature samples. The Diest Formation fills a large erosional channel truncating greensands of different ages, from the upper Oligocene (i. e. about 25 Ma old) to the middle Miocene (i. e. 10 to 15 Ma old) (LAGA, 1973).

## WEATHERING.

Studies on the effect of weathering on glauconites ages are limited in number, especially those dealing with weathering under natural conditions (ALLEN et al., 1964; ODIN et al., 1974b; ODIN and HUNZIKER, 1974; CLAUER, 1976; TRIAT et al., 1976). From this limited number of investigated cases, it would appear that radiogenic strontium is lost preferentially to rubidium, while K and Ar are lost in similar proportions. A similar behaviour is observed for biotite (CLAUER, 1978). However, in case of severe weathering under tropical conditions a lowering of the K-Ar apparent age, up to 20%, may be observed (the glauconites showing this have also been reworked, the latter process being probably without influence on the K-Ar system) (ALLEN et al., 1964; ODIN and HUNZIKER, 1974).

Some published and unpublished data on the greensands from New Jersey are not in conflict with the considerations above (MONTAG and SEIDEMAN, 1981; PASTEELS and KEPPENS, in preparation). In the first paper Rb-Sr apparent ages considerably superior to the probable age of sedimentation are interpreted in term of reworking or alternatively, of preferential loss of Rb versus Sr rad upon weathering. The first interpretation is more probable, since our own data, on samples from the same formation or other ones in the same area, univocally point to a preferential loss of Sr rad upon weathering. To conclude on this point, weathering may cause a lowering of the Rb-Sr age and, in some extreme cases, of the K-Ar age too.

No clear-cut case of weathered glauconite collected in Belgium and adjacent area for dating purpose can be reported here. However, mild weathering may remain unnoticed and collecting samples in fresh outcrops or bore-holes does not prevent from ancient weathering. This is the case namely for the Neogene. This case (except for the Diest Formation previously discussed) will be considered separately, because besides weathering, reworking and daughter nuclide inheritance are possible.

## THE NEOGENE IN THE BELGIAN BASIN.

Our geochronological investigation of glauconite and the adjacent countries started with the "Oligo-Miocene" (ODIN et al., 1974a), in fact the Miocene, according to present-day biostratigraphic interpretations. The data showed poor internal consistency. Considering moreover the biostratigraphic uncertainties, only very prudent conclusions could be drawn. A re-examination of this case is thus necessary.

The Belgian Neogene is to be regarded as an unfavourable case. Transgressions at the Southern edge of the North Sea were of short duration, and most of the time of limited extension. Periods of time where glauconite formation was possible were therefore limited. Greensand deposits are observed, resting above each other, a favourable case for reworking.

The alteration of glauconite to goethite is more or less general, except in the region of Antwerp. It is generally ascribed to subareal weathering in late Tertiary times. Decalcification is also frequent.

These characteristics are reminding of the classical New-Jersey-Maryland coastal plain greensands, which also represent a difficult case for geochronology. As mentioned above, lack of precise biostratiphic data may add to the difficulty.

In the case of the Edegem Sands a considerable spread of apparent ages is observed, from 22 to 31 Ma (with the presently accepted decay constants, STEIGER and JAEGER, 1977), the K-Ar ages being consistently lower than the corresponding Rb-Sr ages. The analyzed glauconites have rather open lattices and K contents varying from 5,7 to 6 % which corresponds to about 15-20% expandable layers. A lower Miocene age (i. e. of about 20 Ma) is probable on the base of nannoplankton assemblage (MARTINI and MUELLER, 1973) and benthonic Foraminifera (DE MEUTER and LAGA, 1976). A former stratigraphic interpretation, pointing to an Oligocene age, and based on planktonic Foraminifera (HOOYBERGHS and DE MEUTER, 1972) is being reinterpreted by the same authors as actually corresponding to the Early Miocene, taking into account the presence of reworked forms from the Oligocene (HOOYBERGHS, pers. comm. to one of us. E. KEPPENS, 1981).

Weathering does not seem to account for the discrepancies observed. Contrary to the rule for the Neogene greensands from Belgium, goethite formation is not observed or then, to a limited scale. The only sample (G. 417) showing signs of oxydation on the glauconite grains (ODIN et al., 1974) and in the outcrop (DE MEUTER et al., 1976) is also the only one yielding more or less concordant ages which may be close to that of sedimentation. Thus those apparent ages (22 Ma with K-Ar, 24 Ma with Rb-Sr) must be regarded with some caution. Finally, the decalcified Kiel Sands resting above the Edegem Sands also contain glauconite. It has been dated and yield a similar discrepant age pattern as most of the Edegem Sands glauconites. Decalcification had thus no effect, apparently at least on the Rb-Sr and K-Ar systems.

Inheritance of daughter isotopes is possible though not previously recorded in glauconites with such relatively high K content. However, there is probably much to be learned still on this subject. A glauconite pellets population may reveal itself highly hererogeneous, with a substantial part of the pellets consisting of disordered "immature" material (VELDE, 1976).

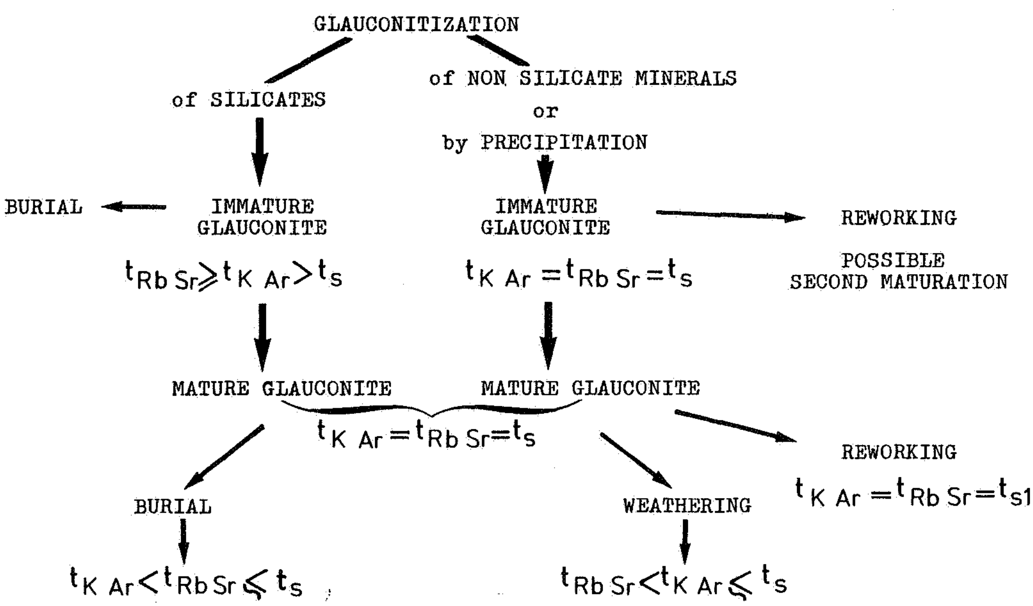
Reworking of part or all the glauconite is also to be considered. In the present case, Upper Oligocene greensands equivalent to the Voort and Boncelles Sands are likely candidates as a source for the reworked material. Since they are approximately 25 to 30 Ma old, a lowering of the K-Ar ages is corollary to such assumption. It may be speculated that a preferential uptake of K, as compared to Rb, in the new depositional environment, may have contributed to the observed discrepancies. This would appear, in any case, more probable than argon loss.

## CONCLUSIONS.

Of all cases investigated in Belgium and North France, only that of the Neogene remain to be elucidated, though there are some clues as to the possible or probable causes of the discrepant age patterns observed. Selection of samples suitable for establishing the age of sedimentation is possible. Similarly, cases of reworking, weathering, probable incomplete glauconitization of silicate material may be investigated with the geochronologic methods in order to determine what their influence is on apparent ages. Clearly more such data are needed. Cases where weathering, reworking, etc... have acted or may have acted simultaneously remain of very difficult interpretation at this stage and should be left over for the time where the effects of all different agencies considered here will be better known. This would require, not only more radiometric data in not too complicated cases, but also additional geochemical, sedimentologic and mineralogical data on the same cases.

At the present, only a very tentative picture can be drawn of the influence of different agencies on the glauconite apparent ages. It is represented in Fig 2.

TENTATIVE SYNOPSIS OF GLAUCONITE AGE INTERPRETATION.




$t_s$ time of sedimentation

$t_{s1}$ time of the first sedimentation in the case of reworked glauconite

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