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Radiometric datation of sediments

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ABSTRACT. — Sediments are poor radioactive clocks. Only in exceptional cases will sedimentation or early diagenesis erase the isotopic memory of earlier events. Diagenesis at low temperature may disturb the isotopic equilibrium of the sediments. Consequently, only in special cases can we attempt to date sediments with radioactive methods. Materials suitable for datation include whole-rock shales, and clay minerals occurring as isolated flakes or pellets. Rb-Sr and K-Ar methods can be utilized on whole rocks and clay-size fractions. Glauconite is a valuable geochronometer (Rb-Sr and especially K-Ar), if the samples are selected on the basis of their high K content and absence of burial and weathering.

Key words: radiometric geochronology, sediments, glauconite, Rb-Sr, K-Ar.

RIASSUNTO. — I sedimenti non sono eccellenti orologi radioattivi. Solo in casi eccezionali la sedimentazione o la diagenesi precoce cancellano il ricordo isotopico degli eventi precedenti. La diagenesi a bassa temperatura può disturbare l'equilibrio isotopico dei sedimenti. Da tutto ciò consegue che la geocronologia radiometrica di sedimenti è fattibile solo in casi eccezionali. Sedimenti argillosi e minerali argillosi (sia in lamelle isolate che in pellets) possono essere utili per datazioni radiometriche. I metodi Rb-Sr e K-Ar possono essere utilizzati sia su roccia totale che sulle frazioni argillose di sedimenti. La glauconite è un utile geocronometro (Rb-Sr e, soprattutto, K-Ar), purchè vengano scelti campioni ricchi in K e privi di alterazioni.

Parole chiave: geocronologia radiometrica, sedimenti, glauconite, Rb-Sr, K-Ar.

The earliest attempts to date sediments with the geochronologic methods based on radioactivity date back from the time when these methods were still in infancy. Natural radioactivity and its possible applications became indeed a subject of interest for earth scientists very soon after its discovery. For example, the observation of the high radium content of deep-sea sediments was made very few years after this new element was discovered (JOLY, 1908). But only years after did this observation lead to the development of a new method of dating sediments.

There are two types of application of radiometric dating methods to sedimentary materials: those based on short-livel radioisotopes, either intermediate members of the natural decay chains, or cosmogenic nuclides on the one hand, and those based on long-lived radionuclides on the other hand. The observation of a high radium content in deep-sea sediments is in relation with the first type of application, which shall however not be considered further here. From methodological and fundamental points of view, dating Quaternary or older deposits are indeed very different problems and only the latter question will be the subject of the present short review.

If we restrict thus our attention to relatively old sediments and sedimentary rocks, we note that in this field as in general the progresses of geochronology based on radioactivity were very slow in the beginning. Before world war II, there is little to be mentioned in this domain, except the attempt to date the Swedish Kolm by the U-Pb method (NIER, 1939).

From this early beginning it became apparent that dating sediments with the radioactive methods is no simple task. Contrary to the case of high temperature systems, the constitution of which corresponds to resetting to zero and the starting at zero of the geochronometer, sediments are poor radioactive clocks. Only in exceptional cases will sedimentation or early diagenesis erase the isotopic memory of earlier events. Sediments undergo moreover diagenetic changes at low temperature, which may disturb their isotopic equilibrium.

The consequences of this are that only in selected cases can we attempt to date sediments with the radioactive methods and that in many instances some degree of imprecision must be tolerated.

Since the early fifties, the potassium-argon and the rubidium-strontium methods became applicable to sedimentary materials and are still the most widely used in this case (INGHRAM et al., 1950; GENTNER et al., 1953; WASSERBURG et al., 1956; LIPSON, 1956, 1958; CORMIER et al., 1956).

Materials suitable for datation include whole-rock shales, clay minerals presenting themselves as isolated flakes, or in pellets as in the case of glauconite (or glaucony to follow the recommendation of ODIN and DODSON, 1982), in different types of sediments.

Datation of shales with the whole-rock rubidium-strontium method

Inasmuch as exchange of strontium isotopes with sea water or sediment water shortly after deposition actually occurs, the Rb-Sr method is in principle applicable to shales. Argon being a noble gas, such equilibration is unexpected for its isotopes, so that attempts to fix the sedimentation time with the K-Ar method using whole-rock samples would make little sense.

Among the main contributions to the subject of dating shales, we may mention: COMPSTON and PIDGEON (1962), WHITNEY and HURLEY (1964), COMPSTON et al. (1956), PETERMAN (1966), CHAUDURI and BROOKINS (1970), CLAUER (1973, 1976), PERRY and TUREKIAN (1974), SPEARS (1974), CHAUDHURI (1976).

The basic condition (besides that of closed-system) for the application of the Rb-Sr isochron method is the availability of several samples with different rubidium to strontium ratios which all contained a strontium with the same isotopic composition at the initial, i.e. sedimentation time. It is important to observe that both conditions are to be satisfied simultaneously, since some factors which may cause the elemental ratio to vary, may cause the isotopic ratio to vary also. In addition, the repartition, a straight line of experimental points, does not prove that both conditions above are satisfied since the mixture in variable proportions of two components with different Rb/Sr and $^{87}Sr/^{86}Sr$ ratios also results in a straight line repartition of experimental points, which is without chronological significance in that case. The investigation of weathering profiles and recent sediments shows an almost complete lack of strontium isotopic equilibration during weathering and oceanic or estuarine sedimentation (DASCH, 1969; BISCAYE and DASCH, 1971; BISCAIE, 1972). It has been shown however by CLAUER et al. (1976) that isotopic exchange actually occurs, at least in some cases, but after sedimentation and progressively. A recent review of this problem is made by CLAUER (1982). Isotopic equilibration shortly after sedimentation is only one of the different situations which are actually observed. The rate of this equilibration depends on mineralogy and grain size, among other factors. Detritic clays may fail to have reached isotopic equilibration with sea water, sediment water or other components of the sediment, more than 50 Ma after deposition.

The proposal of CORDANI et al. (1978) should also be considered with some caution. They argue that some degree of isotopic homogeneisation can be achieved by mechanical mixing of detritic, more or less weathered, diagenetic, more or less equilibrated components, inasmuch as similar depositional conditions prevail over a wide area. If isotopic equilibration is attained by such a process, some degree of chemical homogeneisation is likely to occur also. The case becomes impossible to solve by the Rb-Sr method because of too similar Rb/Sr ratios. Alternatively, a collection of samples with Rb-Sr ratios which differ widely will generally present a wide range of mineralogical compositions. Carbonate-rich samples with low Rb-Sr ratio will tend to have lower initial $^{87}Sr/^{86}Sr$ ratios than silicate-rich ones.

Several cases have been reported where diagenesis (or « weak metamorphism ») is considered the cause of isotopic rehomogeneisation (BOFINGER et al., 1968, 1970; CORDANI et al., 1978; PERRY and TUREKIAN, 1974; BONHOMME et al., 1982, etc.). A «true isochron» or a not too scattered «errochron» will then give the age of the diagenesis, or a good approximation of it.

We shall not consider here the possible «disturbing factors» which may cause the experimental points to be widely scattered on the isochron diagram. In this case, it is indeed obvious that no age can be derived from the data. The important point is the possible existence of « false isochrons » yielding spurious age indications, or true isochrons, giving the age of another event than that at which the attempt at datation is directed (in casu, the sedimentation time).

This second possibility is not to be ruled out a priori owing to the possibility of a slightly positive or (less likely) negative initial slope for the isochron, or to late diagenetic evolution. From this may result, even in the general case, a variable error, which may amount to several Ma or to several tens of Ma. Thus, for some applications, as for example the calibration of the time-scale for the Phanerozoïc, whole-rock Rb-Sr data on shales should be used only with great caution. For the deciphering of unmetamorphic Precambrian terranes however they may reveal themselves very useful.

K-Ar dating on whole-rock samples and size fractions of shales

K-Ar measurements on whole-rock and size fractions, the mineralogical composition of which, being determined by X-rays, may help the interpretation of the Rb-Sr dates, or provide information on the source of the detritic components or the diagenetic evolution of the sediment (HOFMANN et al., 1974; PERRY, 1974). In the same rock components may be found which keep record of their pre-depositional history and others recording the last diagenetic event. Though Rb-Sr systems may react differently, these observations tend to support the idea that the determination of an age which approximates that of sedimentation, by the Rb-Sr method, may in some cases result from a kind of averaging of the ages of pre- and post-depositional events.

Fine fractions

The Rb-Sr and K-Ar methods may be applied to the clay-size fraction of the sediment. In such cases the detritic components or at least a large part of them may be eliminated. Moreover the mineralogic investigation of the clay helps greatly to the interpretation. Reviews of this question have been published recently by the main contributors to the development of this method (BONHOMME, 1982; CLAUER, 1982). They report cases were early diagenesis could be dated on low temperature illite. However, in other instances detritic 2M illite predominates and yields the approximate age of the source. In other sediments or sedimentary rocks the presence of 2M illite reflects an anchimetamorphic evolution and the only event which can be dated is a certain stage of this evolution. Finally, the presence of kaolinite denotes alteration by weathering which helps to explain the scatter of experimental points on an isochron diagram.

Very clearly, the «ages of sedimentation» (or close to sedimentation) which can be obtained this way are much more precise and established on firmer ground than when whole-rocks are chosen as material for datation, in the absence of mineralogical study. The variety of situations observed also shows that there is no miracle to be expected which would allow the precise dating of sediments when no attempt is made at the elimination of the detritic components and when the composition of the clay fraction (which may be detritic, early or late diagenetic) remains unknown.

Glaucony

The nature of glaucony or glauconite

In the present case, it seems more important to define as precisely as possible the material *which is actually submitted to analysis* when geochronologists decide to « date glauconite » than to give a precise definition of a mineral species, or a group of minerals. The use of the term « glaucony » with the same definition as in ODIN and DODSON (1982) and CLAUER (1982) is quite convenient for this purpose. The word glaucony will thus be used as a facies name in order to avoid confusion with a peculiar mineralogical composition.

Glaucony presents itself in the pelletal form in sediments in most instances and what is thus analyzed are these pellets, which may be plurimineral, in some instances at least. However, in most cases the essential (or only) constituent of the glaucony pellets is a member of the family of « glauconitic minerals », an evolutive series starting from a smectitic protoglauconite and ending with the K-rich illitic member (mineral glauconite or glauconitic mica), which can be defined as an iron illite polymorph. The intermediate terms are mixed-layered minerals. Besides the common pelletal form, glauconitic minerals may be observed as flakes dispersed within a sediment or as a coating on pebbles or hard-grounds.

Formation of glauconitic minerals

The question of glauconite formation has been much disputed in the past. From a number of rather recent studies, a few points would appear which can be summarized briefly (EHLMANN et al., 1963; GIRESSE and ODIN, 1973; GIRESSE, 1975; LAMBOY, 1968, 1975; ODIN, 1973, 1975; ODIN and MATTER, 1981).

a - Glauconitization, i.e. the formation of glauconitic minerals, does not require a specific precursor, as for example detritic clays or micas, but a suitable microenvironment or substrate. This microenvironment is « semi-confined »: ion exchange with sea-water is possible but Eh and pH conditions may be slightly different from that of sea water. The ideal size of this substrate depends its porosity. Coproliths, foraminiferal skeletons, detrital mica flakes are among the most commonly observed substrates, evolving into glauconite grains the shape of which, at early stage of development, is indicative of the nature of the initial substrate. At stage, the size of the initial grain may increase and its morphology be altered. The original clay minerals, mica, carbonates being then largely or totally eliminated, the glauconite pellet itself becomes the substrate for further glauconitization.

b - Within the substrate, the smectitic end-member appears, seemingly generated by ionic chemical precipitation. Simultaneously, the pre-existing minerals (clay, mica, carbonates, but also quartz, feldspar...) are « replaced », i.e. start to disappear. This occurs at variable rates, depending on the initial mineral. Carbonates, for example, are more easily replaced than silicates or quartz.

c - By potassium uptake the smectitic end-member progressively evolves towards the illitic end-member. A further elimination of the substrate takes place. In some instance the elimination occurs mainly at this stage.

d - Individual glaucony pellets may consist out of a mixture of low-K and high-K glauconitic minerals (VELDE, 1976).

e - This evolution comes to an end when ion exchange with sea water is no longer possible, because of burial. Alternatively, a mature glauconite is generated before burial, and the evolution cannot proceed further. Glauconite levels are indeed often associated with lowered sedimentation rates or unconformities.

Zero isotopic age of glauconite

It is quite evident from the description above of the processes of glauconite formation that an « isotopic memory » of the silicate precursor may be kept by the glaucony pellet as long as a maturation stage is not reached where this precursor is totally eliminated. The question has been recently reviewed by ODIN and DODSON (1982). Several cases are discussed by them, showing convincingly that immature pellets with about 1 % K may yield apparent K-Ar ages up to 100 Ma higher than the time of sedimentation. Whenever glauconite suites are found at the same locality or in the same formation, showing different stages of maturation, the «excess argon» sharply decreases with increasing K content. At about 5 % K (6 % K_2O) the isotopic memory of the precursor seems to have been erased. When the substrate consists of carbonate debris, low-K glauconites may yield ages close to sedimentation. An «isotopic memory» of the precursor may however be present in cases where X-ray diffractometry fails to show the presence of other minerals than glauconite minerals in the pellets.

These K-Ar data on « immature » glauconite have been recently complemented by a Rb-Sr investigation of the same samples. The apparent ages found by Rb-Sr are similar or slightly older than the corresponding K-Ar ages. In other words, glauconies carrying excess radiogenic argon also carry excess radiogenic strontium and this in similar or slightly higher proportions (KEPPENS et al., 1984). It has also been observed (KEPPENS and O'NEIL, 1984) that in suites of samples of the same age, high-K glauconies have higher $\delta^{18}O$ values than low-K glauconies. The latter case would correspond to incomplete equilibration with sea-water oxygen.

Other cases than those selected by ODIN and DODSON (1982) may be found in the literature which are evidently to be interpreted in terms of argon or strontium excess present in low-K glauconite (e.g. OWENS and SOHL, 1973; PASTEELS and KEPPENS, 1981). In the second mentioned paper, a suite of glauconies from the Cretaceous yield concordant Rb-Sr and K-Ar ages except in one case where the Rb-Sr age is higher. This particular sample has a lower K content (5.2 %) than all the others. From its stratigraphic position and other data allowing the calibration of this part of the time-scale, it would appear that the K-Ar ages are (at least approximately) correct, the Rb-Sr ages some 10 Ma too high.

Influence of burial and heating on glauconite ages

Few systematic investigations have been undertaken on the influence of diagenesis sensu-stricto (anadiagenesis) or deep diagenesis (anchimetamorphism) on glaucony K-Ar ages (FREY et al., 1973; CONARD et al., 1982; FISCHER et al., 1984). In the Helvetic Alps, a high K glaucony starts to lose radiogenic argon before the stilpnomelane isograd is reached (FREY et al., 1973). Many published data, namely on samples from bore-holes, clearly show that burial diagenesis causes argon loss from glaucony, but precise information on the diagenetic conditions in which this occurs are generally lacking.

The problem has also been considered from the experimental point of view. Glauconies of different K contents were subjected to hydrothermal treatments during periods of time up to three months. The mica-type glauconite loses from 4 to 8 % of its argon ar 320° C, but K-poorer, mixed layered glauconitic minerals are less retentive in the same conditions (ODIN et al., 1977; ODIN, 1982).

Corresponding Rb-Sr data are still much more scarse and inconclusive. It has been argued, on the basis of the cases where a comparison is available, that Rb-Sr ages might be less influenced than K-Ar ages by burial diagenesis (KEPPENS and PASTEELS, 1982) but this point needs confirmation.

Influence of weathering on glauconite ages

The K-Ar and Rb-Sr investigation of the same in-situ and reworked, weathered glauconies in Normandy tends to show that, upon weathering, glauconies release more easily radiogenic strontium than radiogenic argon (ODIN and HUNZIKER, 1974; CLAUER, 1976).

Table 1 presents other data bringing a firm confirmation to this, in the case of a suite of samples from the New Jersey-Maryland greensands. The K-Ar data are taken from the paper of OWENS and SOHL (1973). The Rb-Sr ages have been measured in Brussels (VUB). The labelling of the samples 1, 2, 3 is that of the above-mentioned publication and refers to the material used for K-Ar dating. The corresponding numbers with an asterisk refer to the glaucony used for Rb-Sr dating which result from another mineral separation from the same sediment sample. Most of these greensands show evidence of alteration by weathering, as described by WOLFF (1967). For lack of a better criterion the abundance of goethite was taken as an indication of the intensity of weathering. In samples 3, 5, 6 some goethite pseudomorphs after glauconite are observed which were carefully removed from the analyzed material. Samples 12 and 13 consist of glaucony pellets embedded in a

TABLE 1

New Jersey-Maryland greensands. Comparison of geological and radiometric data

Stage	Probable age of sedimentation	Sample No.	t_{K-Ar} (Ma)	t_{Rb-Sr} (Ma)	δ_{K-Ar} (1)	δ_{Rb-Sr} (1)	Weathering
Ypresian	50-56	1 1*	55.7 ± 1.8	52.3 ± 2.3	0	0	unsignificant
Thanetian	58	3 3*	58.5 ± 1.9	52.5 ± 1.7	0	−5-10	moderate
Danian	63	5 5*	61.3 ± 2.0	56.8 ± 1.5	0	−5-10	moderate
	63	6 6*	63.4 ± 2.1	54.3 ± 1.0	0	− 15	moderate
		Probable	unconformity				
Lower Maestrichtian or	73	12 12*	62.5 ± 2.1	57.5 ± 1.4	−10-15	−20	pervasive
Upper Campanian		13 13*	61.4 ± 2.0	56.3 ± 2.5	−10-15	−20-25	pervasive
Campanian	75	22 22*	74.2 ± 2.4	69.6 ± 1.9	0	−5-10	unsignificant

(1) δ: percent departure from probable true age.

goethite matrix. Again, the goethite was removed and only fresh-looking glauconies were analyzed. If we compare the K-Ar and Rb-Sr ages with the stratigraphic age we observe that only samples 12 and 13 show argon loss, while a lowering of Rb-Sr ages is much more important in the same cases and also occurs for what would appear at first sight « slightly weathered » glauconite. These data also show that radiogenic strontium may be lost in cases where, apparently, no argon is lost.

Concluding remarks on the usefulness of glauconite as a geochronometer

If selected on the base of high K content, absence of burial and weathering, glauconite may prove a valuable geochronometer with the K-Ar and Rb-Sr methods, especially the former.

Comparison of Rb-Sr and K-Ar dates may help interpretation since radiogenic strontium is more easily lost than radiogenic argon upon alteration by weathering, and in the case of immature glauconies it would appear that « inherited » radiogenic strontium may be less easily eliminated than radiogenic argon.

A subsisting question is that of the degree of precision which can be obtained in the most favourable case. This problem can be solved by the analyses of many samples from the same stratigraphic level which are relatively young in order to evidence small differences in radiometric ages. Such study has been carried out on late Oligocene glauconies by KREUZER et al. (1980). The analytical errors only cannot account for the scatter of the results they observe. Therefore a « principle error in the glauconite system » should be considered which is « at least of the order of several hundred thousand years » (ref. cit.). This combines the « zero age » uncertainty with the possible alteration of the parent-daughter system its formation. Another factor which may contribute to this principle uncertainty is the principal difference between age of formation and age of deposition. Many glauconites are probably not formed where they are found but transported over short distances before final deposition (it should be kept in mind however that fossils can be transported in the same way too).

This « principle error » restricts the use of glauconite to samples older than the late Tertiary. It remains nonetheless among the datable outhigenic minerals, that which is the easiest to identify and recover from sediments, thus probably also the most useful.

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