

Uranium–thorium–lead age-determinations with respect to the Phanerozoic time-scale

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SUMMARY

Topics discussed in this short review are: the physical basis of the method, common lead ages, calculation of results, the significance of discordant results, and the selection of material for analysis. It is stressed that all aspects of the geology of an area, including its mineralogy and geochemistry, must be studied before age-determinations on material can be properly evaluated.

1. Introduction

AGE-DETERMINATION by means of uranium, thorium, and lead has been discussed in many original papers, review articles, and books in recent years. The aim of this review is to emphasize features of especial concern to stratigraphers. The accompanying references list some of the more important recent papers concerning uranium–thorium–lead methods, through which earlier references can be traced, and also several textbooks containing more detailed background information on various related topics.

2. The basis of the method

Naturally occurring uranium consists of two isotopes, ^{238}U and ^{235}U , with relative abundances at the present time of 99.27 per cent and 0.72 per cent respectively. Both these isotopes are unstable and give rise to radioactive decay series which have as their end-products ^{206}Pb and ^{207}Pb . In the course of decay from ^{238}U to ^{206}Pb there are 14 intervening unstable nuclides; from ^{235}U to ^{207}Pb there are 12. Of the two parent nuclides, ^{235}U decays more rapidly, any given amount halving itself in 713 m.y., whereas ^{238}U has a longer 'half-life' of 4510 m.y. Since ^{235}U decays more rapidly than ^{238}U the ratio $^{235}\text{U} : ^{238}\text{U}$ has decreased throughout geological time down to the present-day relative abundance of 1 : 137.7. Naturally occurring thorium consists of a single isotope ^{232}Th with a half-life of 14 200 m.y. There are 10 intervening nuclides between ^{232}Th and its stable end-product ^{208}Pb .

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The decay of an atom of ^{238}U to its final end-product involves the production of 8 alpha particles (helium nuclei); similarly the complete decay of an atom of ^{235}U produces 7 helium nuclei, and ^{232}Th produces 6. Thus both uranium and thorium differ from the other two elements of interest to geochronology, namely potassium and rubidium, in that their radioactive decay passes through a large number of intervening stages. Decay-products begin to accumulate from the moment of formation of a radioactive mineral, but, as there is a series of unstable isotopes with varying half-lives, there is a time-interval before one disintegration of a parent atom is accompanied by one disintegration of each successive decay product. When this occurs the whole series is said to be in equilibrium. The time required for equilibrium to be reached by uranium minerals is about a million years, but by thorium minerals only about 30 years. It is possible to use radiometric equipment to determine whether a uranium mineral is in equilibrium, since the radioactivity of natural uranium (or thorium) is contributed by both the parent element and intermediate decay-products, each with β - and γ -radiation in different proportions. For a decay series as a whole the proportion of β - to γ -radiation is fixed as long as it is in equilibrium, and since radiometric counting equipment can be used to count γ - and $\beta + \gamma$ -radiation separately the state of equilibrium can be determined. Uranium that has only recently migrated to its present position, or uranium that has recently lost some of its decay-products can be readily distinguished in this way. This is a convenient means of eliminating at an early stage of an investigation material that is unsuitable for age-determination.

For any decay series in equilibrium the relationship between the number of parent atoms (P) at the present time and the number of daughter atoms (D) produced in time t is given by

$$t = \frac{1}{\lambda} \ln \left(1 + \frac{D}{P} \right),$$

where λ is the decay constant of the parent isotope, which is related to the half-life $T_{\frac{1}{2}}$ as follows:

$$T_{\frac{1}{2}} = \frac{\ln 2}{\lambda} = \frac{0.6931}{\lambda}$$

This relationship covers the decay of ^{238}U to ^{206}Pb , of ^{235}U to ^{207}Pb , and of ^{232}Th to ^{208}Pb , and enables a reliable age to be derived from any of these pairs, provided there has been no loss or gain of parent or daughter isotopes. Because of the different rates at which ^{235}U and ^{238}U decay, the ratio of the end-products ^{207}Pb and ^{206}Pb is itself a measure of age. Fig. 1 shows the values of the ratios N_{206}/N_{238} , N_{207}/N_{235} , N_{207}/N_{206} , and N_{208}/N_{232} over the period 0 to 600 m.y., where N is the number of atoms of the various isotopes. The diagram illustrates the uncertainty in the determination of the decay constants, and also the sensitivity of the ages to changes of one unit in the third significant figure of the ratios. It can be seen that the N_{206}/N_{238} ratio has negligible uncertainty and a satisfactory slope, whereas N_{207}/N_{206} is unsatisfactory in both respects. In addition to these two criteria others also enter into assessing the relative reliability of the different ratios, in particular the absolute amount of each lead isotope present. The most abundant isotope can be determined

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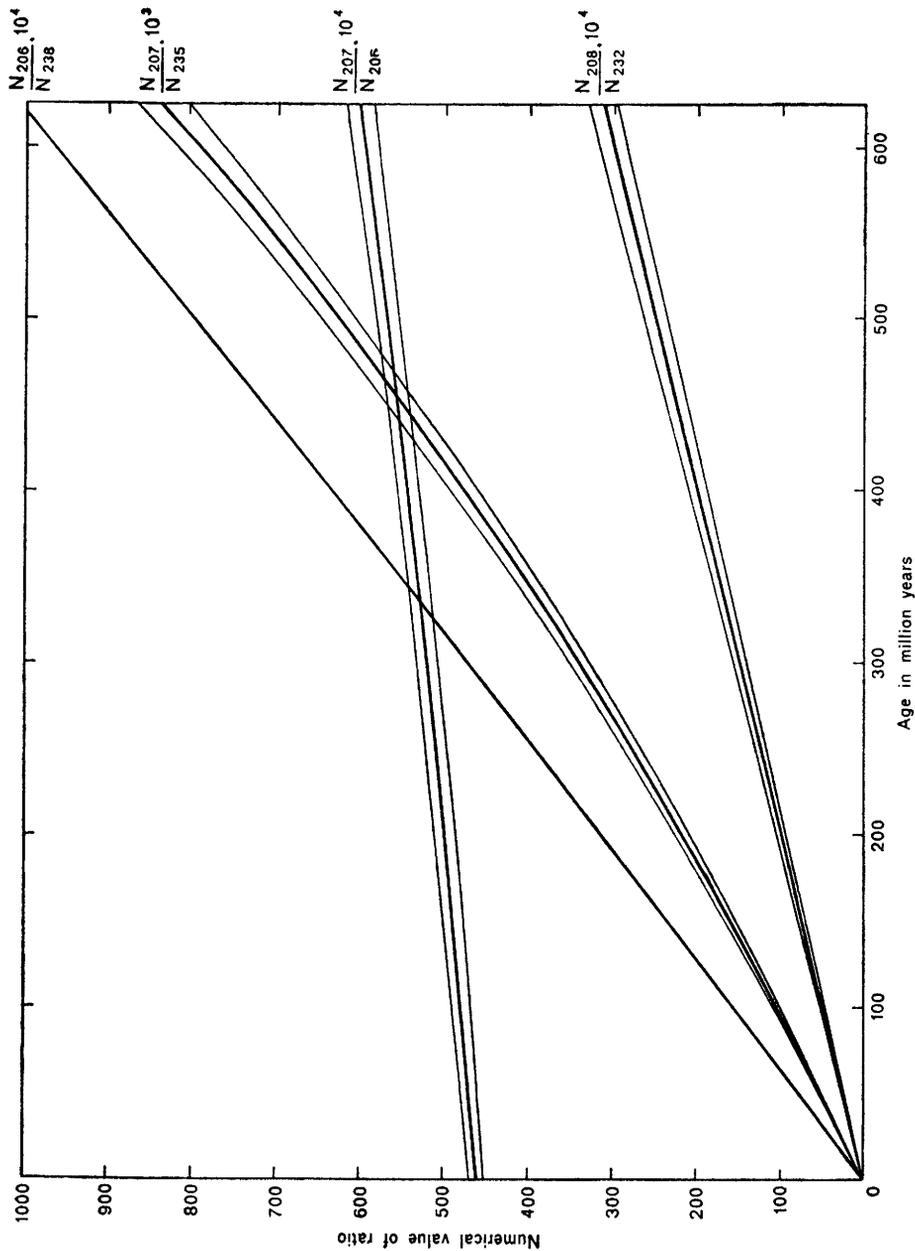


FIG. 1. Numerical values of various ratios plotted against ages (in millions of years). The central line shows the mean value of the ratio; the outer lines show the limits of uncertainty of ratio owing to the uncertainty with which decay constants are known. Note that for N_{206}/N_{238} the uncertainty is within the width of the line.

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with the greatest relative accuracy and is least sensitive to the common-lead correction (discussed below). For a uranium-bearing mineral that crystallized in the period 0 to 600 m.y., ^{206}Pb constitutes about 95 per cent of the uranium-lead formed, ^{207}Pb only about 5 per cent. Thus ^{206}Pb can be determined with much greater precision than can ^{207}Pb , particularly in young materials where the total amount of lead present is small. This fact enhances the reliability of the N_{206}/N_{238} ratio and diminishes that of N_{207}/N_{235} and especially N_{207}/N_{206} . There is no clear-cut dividing-line below which it can be said that N_{207}/N_{206} ages are completely unreliable; they become progressively less reliable from Pre-Cambrian time onwards, but post-Palaeozoic ages are almost invariably unreliable.

Ages based on the ratio N_{206}/N_{210} , where N_{210} is the number of ^{210}Pb atoms, merit consideration. ^{210}Pb is an unstable intermediate nuclide in the decay of ^{238}U to ^{206}Pb . Since for a decay series in equilibrium the quantities of intermediate decay members are proportional to the amount of the parent remaining, the ratio of any intermediate member to the stable end-product is related to the age of the system. Comparatively few N_{206}/N_{210} ages have been reported, although the measurement of N_{210} can be carried out radiometrically. Both N_{210}/N_{206} and N_{207}/N_{206} ages (sometimes known as lead-lead ages) can be obtained without determining the total uranium, thorium, or lead content, since it is necessary to determine only the isotopic composition of a separated portion of lead.

3. Common lead ages

Before continuing the discussion of uranium-thorium-lead methods of age-determination applied to radioactive minerals it is necessary to consider average crustal lead, which consists of four isotopes ^{204}Pb , ^{206}Pb , ^{207}Pb , and ^{208}Pb . ^{204}Pb is not derived from any radioactive parent so far as is known. It is generally accepted that the composition of average lead at the time of the Earth's formation was the same as that now found in those meteorites that have a negligible uranium and thorium content, such as the Cañon Diablo meteorite, which has a composition of ^{204}Pb 2.0, ^{206}Pb 18.8, ^{207}Pb 20.6, ^{208}Pb 58.6 atomic per cent. Throughout the history of the Earth the composition of the original average lead has changed progressively with the addition of ^{206}Pb , ^{207}Pb , and ^{208}Pb from uranium and thorium also present in the Earth. There is evidence for supposing that the isotopic composition of crustal lead is established at a depth below that at which any exposed igneous rocks were emplaced. Indeed, it is only possible to envisage the continuous homogenization of existing lead with new radiogenic lead at very deep levels in the crust or in the upper part of the mantle. Because this common lead is continuously changing in isotopic composition with time, it is theoretically possible to determine its age. To do this it is necessary to choose four parameters additional to those required for dating uranium and thorium minerals. These cover the composition of original lead and the age of its formation (considered by some authors to be the age of the Earth, by others the age of the crust). Using these basic data the Holmes-Houtermans method of calculation is independent of variations in the U/Th ratio of parent material, whereas other methods are not. This leads to different assess-

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ments, according to the method used, of what constitutes an ‘anomalous’ result, but these are beyond the scope of this review. Russell (1963) provides a more extended introduction to the problems concerned. It will be apparent, however, that ages obtained from common lead differ in certain important respects from the ages derived from the analysis of uranium or thorium minerals. First, there is a wider choice of parameters that can be used in calculating an age, and for purposes of comparison it is important to know which have been used. Secondly, the method is very dependent upon a high level of analytical precision. Thirdly, the age does not date the crystallization of the mineral but (provided it has not suffered contamination from lead of a different composition) the time at which the lead separated from its source. It is possible to find examples of galena where the difference between model age and time of crystallization established from geological evidence is 300 m.y.¹

4. Calculation of results

Most uranium minerals, except those formed at low temperatures, contain some thorium, and most thorium minerals contain some uranium. To obtain an age-determination normal practice is therefore to analyse for both uranium and thorium as well as for lead. Ideally, measurement of these three elements alone is sufficient to calculate the age of a mineral. When this is done by chemical methods this is often referred to as a ‘chemical age’. Wickman (1944) has published graphs that enable a chemical age to be obtained directly from a chemical analysis. The lead-alpha method developed by Larsen and co-workers about 1952, and applied to zircon, monazite, thorite, and xenotime, relies on the radiometric determination of the combined uranium and thorium content, and lead determination by optical emission spectrography. For this method it is necessary to assume the value of the U/Th ratio in any particular concentrate and choose the appropriate constant for calculation of the age.

Both the chemical age and the lead-alpha age may be grossly misleading, since there is no means of knowing the extent to which lead was present in the mineral at the time of its formation, nor is there anything to indicate whether or not a mineral has been a chemically closed system since its formation. Many uranium and thorium minerals contain significant amounts of common lead. In the case of low-temperature pitchblende it may be 50 per cent of the total lead present. This lead may be visible as galena inclusions or it may be invisible at the highest magnifications. Isotopic analysis of the lead present in a mineral determines the percentage abundance of ²⁰⁴Pb, ²⁰⁶Pb, ²⁰⁷Pb, and ²⁰⁸Pb. If the mineral is completely free of common lead, as may happen with high-temperature uraninite, then ²⁰⁴Pb will not be found. If it is present then not all of the ²⁰⁶Pb, ²⁰⁷Pb, and ²⁰⁸Pb has been produced in the lifetime of the mineral and a correction based on ²⁰⁴Pb must be

¹ For example, at Tyndrum in Perthshire, post-Caledonian fault-fissures parallel to the Great Glen fault contain lead, zinc, and uranium minerals that are apparently contemporaneous. The model age of the galena is *c.* 550 m.y. (Holmes-Houtermans model, from table v of Pockley 1961), but the age of pitchblende from the same section of the vein is 230 m.y.

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made. Low-temperature uranium minerals with high common lead contents commonly have a negligible thorium content, in which case all the ^{208}Pb is attributable to common lead. Since the ratio of $^{204}\text{Pb} : ^{208}\text{Pb}$ in common lead varies between about 1 : 33 and 1 : 39 according to its age, a common lead correction based on ^{208}Pb is preferable. The choice of common lead composition used for the correction depends on circumstances. If galena is present in the same specimen or the same vein or horizon that provided the radioactive mineral, then this should be analysed and used for the correction. Failing this, judgment must be exercised in selecting a composition corresponding to that of common lead in the region at the time the radioactive mineral crystallized. However, even in the most unfavourable circumstance of a very young mineral with large common lead contamination, the precise value used for common lead is not critical, since changes in common lead composition with time are small. Of course if there is any abnormality in the composition of the lead incorporated in a mineral at its formation, then the problem of correction becomes much more difficult. Further details of the method of calculation of ages from basic analytical data and tables for the rapid conversion of ratios into ages are published by the United States Geological Survey (Stieff *et al.* 1959).

After an appropriate correction for common lead has been made, and provided the mineral has not been altered, the isotopic ratios should all be in exact agreement, but for reasons outlined below, this seldom happens. If there are discrepancies between the isotopic ages, the term 'apparent age' is sometimes used where there is uncertainty as to the significance of the figures obtained in order to emphasize that the calculated age is not necessarily the true age.

5. Discordant results

The simplest examples of discordance between calculated ages are brought about by loss or gain of uranium or lead through recent supergene action. Appreciable supergene alteration of a high-grade uranium mineral normally results in distinct mineralogical changes both in the mineral itself and its surroundings, so that discordance of the ratios would merely confirm what was obvious. Alteration of low-grade materials may not be so apparent and in these circumstances the degree of concordance may be the only criterion of reliability available. The effect of recent lead loss on the N_{206}/N_{238} and N_{207}/N_{235} apparent ages of uraniumiferous material of various true ages up to 600 m.y. is shown in Fig. 2. Theoretically, an N_{207}/N_{206} age remains unaltered under these circumstances at the true value, whereas an N_{207}/N_{235} apparent age is always greater than that of the corresponding N_{206}/N_{238} age. However, as indicated above, N_{207}/N_{206} ages are unreliable over most of Phanerozoic time, and it is often supposed in these circumstances that the degree of concordance between the other two ratios is a sensitive index of reliability. This is erroneous, for as shown in Fig. 3 discordance reaches a maximum with between 50 and 60 per cent lead loss and subsequently decreases. Furthermore, the maximum possible discordance due to lead loss for material with a true age of 300 m.y. is 10 m.y., whereas the maximum discordance for material with a true age of 100 m.y. is only 1 m.y. In this age range concordance is clearly not a guide to

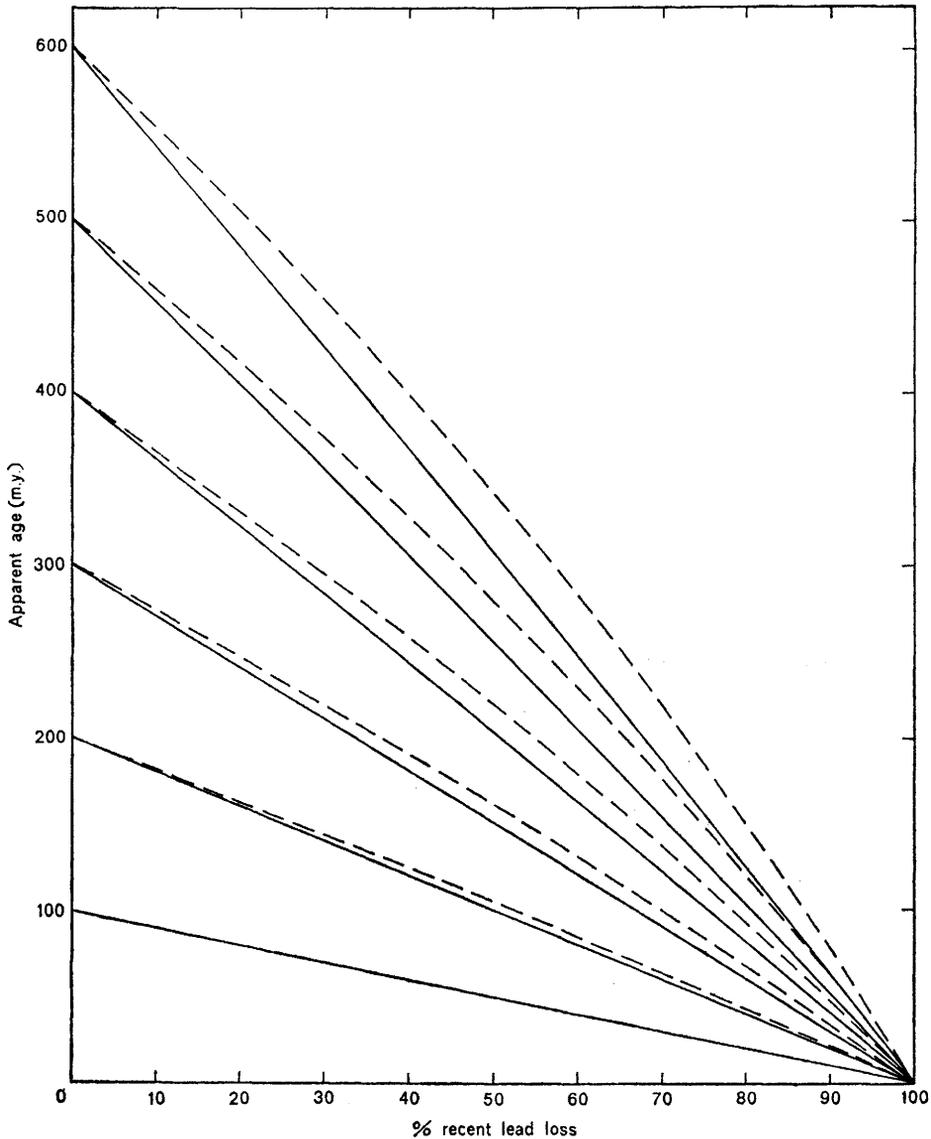
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FIG. 2. Variation of the N_{206}/N_{238} apparent ages (solid lines) and N_{207}/N_{235} apparent ages (dashed lines) with increasing recent lead loss for material of true age 100, 200, 300, 400, 500, and 600 m.y. The curves for material of 100 m.y. true age are coincident.

reliability. The effect of recent uranium loss upon apparent ages is shown in Fig. 4. Again N_{207}/N_{206} is theoretically correct, whereas under these circumstances an N_{206}/N_{238} 'age' is greater than an N_{207}/N_{235} 'age'. Discordance increases progressively with uranium loss as well as with increasing true age (Fig. 5). As an example of the latter trend, a recent loss of 50 per cent uranium produces a discordance of

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about 2 per cent between apparent ages where the true age is 50 m.y., but for the same uranium loss there is a discordance of about 13 per cent where the true age is 200 m.y. Thus post-Palaeozoic (and particularly Tertiary) material can suffer very large uranium losses and show only small discordance.

Discordance between apparent ages is more commonly encountered as a consequence of a mineral not having been a closed system throughout its history, that is, as a consequence of metamorphism or hydrothermal alteration. As with supergene alteration this may involve the relative loss of uranium or lead, but for material of any given true age the degree of discordance diminishes as the age of alteration increases. This is to say that the discordance is less if alteration occurred in the geological past than if it had occurred recently. Aldrich & Wetherill (1958, fig. 10) provide a diagrammatic illustration of this. If a number of discordant analyses of material formed at the same time and altered at a later time are available, a graphical method devised by Wetherill (1956) can be used to obtain both ages. The resulting plot can be readily interpreted only where alteration (addition or loss of uranium or lead) has taken place only once, and where the interval between the formation of a group of minerals or rocks and their alteration is more than about 400 m.y. Thus the method has no practical application to most problems within the Phanerozoic time-scale. It should be mentioned in this context that

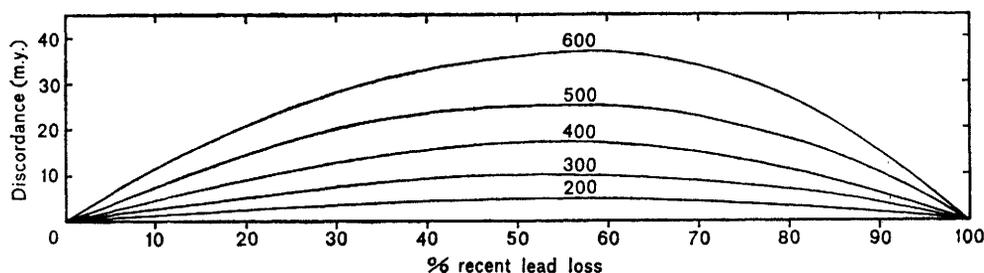


FIG. 3. Graph showing how the difference between N_{206}/N_{238} and N_{207}/N_{235} apparent ages ('discordance') varies with increasing recent lead loss for material of true age 200, 300, 400, 500, and 600 m.y. The curve for material of true age 100 m.y. is not shown, since it almost coincides with the base-line.

loss of lead by continuous diffusion has been proposed as a possible explanation of some discordant ages, but this hypothesis has not yet been accepted by all workers.

Discordance can arise in another way, which applies particularly to finely divided uraniferous material contained in porous sediments. In the decay of ^{238}U one of the intermediate nuclides is ^{222}Rn , a gas with a half-life (3.8 days) long enough to permit it to escape from its environment. Although there are also gases in the decay series of ^{235}U and ^{232}Th , their half-lives are too short for escape to be a significant factor. Loss of ^{222}Rn over a long period of time would reduce the accumulation of ^{206}Pb and give a low $^{206}\text{Pb}/^{238}\text{U}$ age and a high $^{207}\text{Pb}/^{206}\text{Pb}$ age, while the $^{207}\text{Pb}/^{235}\text{U}$ age would be correct. The pattern of discordance is similar to that of lead loss, but in this instance the discordance between N_{207}/N_{235} and N_{206}/N_{238}

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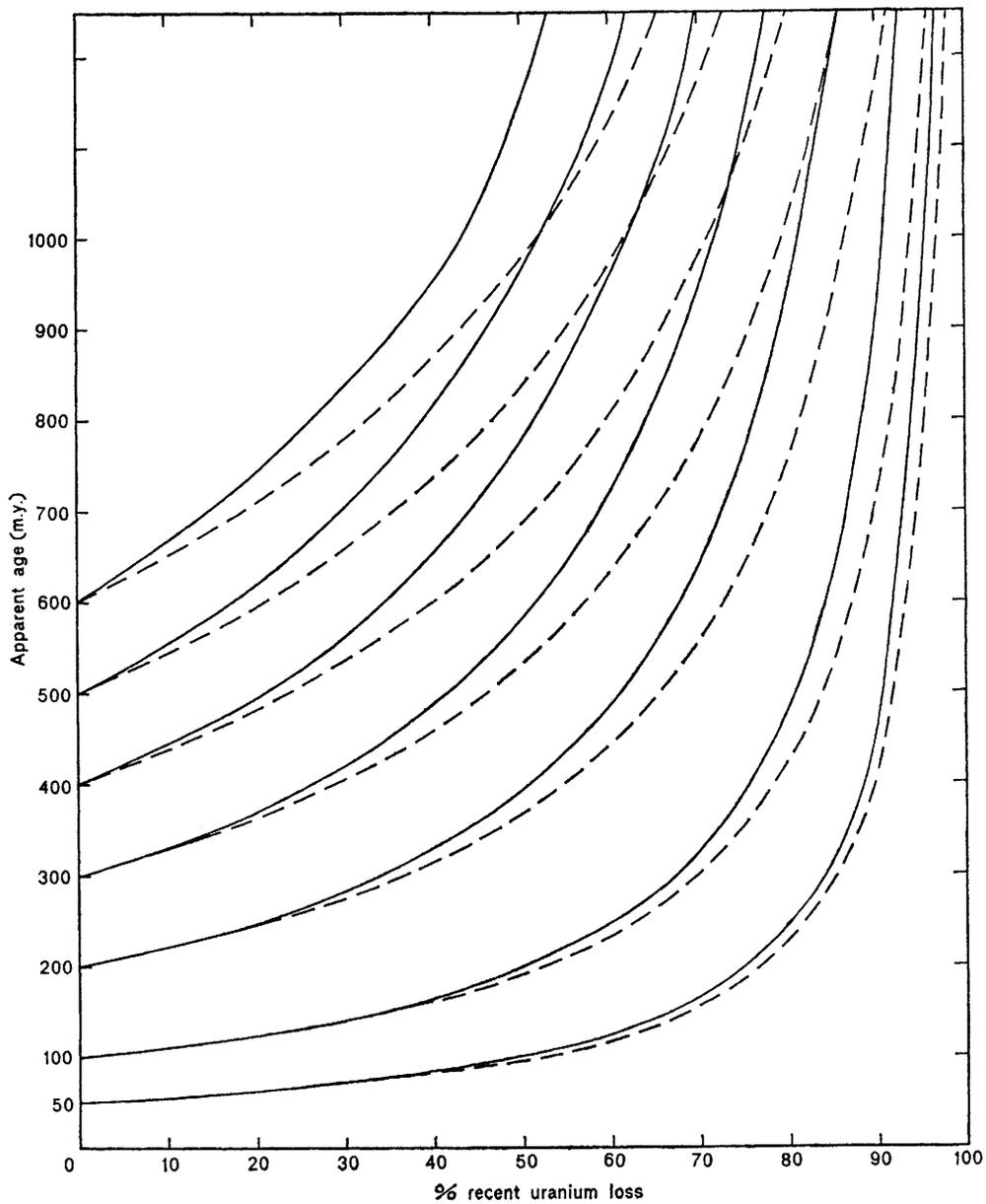


FIG. 4. Variation of the N_{206}/N_{238} apparent ages (solid lines) and N_{207}/N_{235} apparent ages (dashed lines) with increasing recent uranium loss for material of true age 50, 100, 200, 300, 400, 500, and 600 m.y.

ages could theoretically equal the true age of the material. Long and Kulp believe that radon loss combined with lead loss is a significant factor affecting the apparent ages of the bedded uranium ore deposits of the Colorado Plateau. Small quantities

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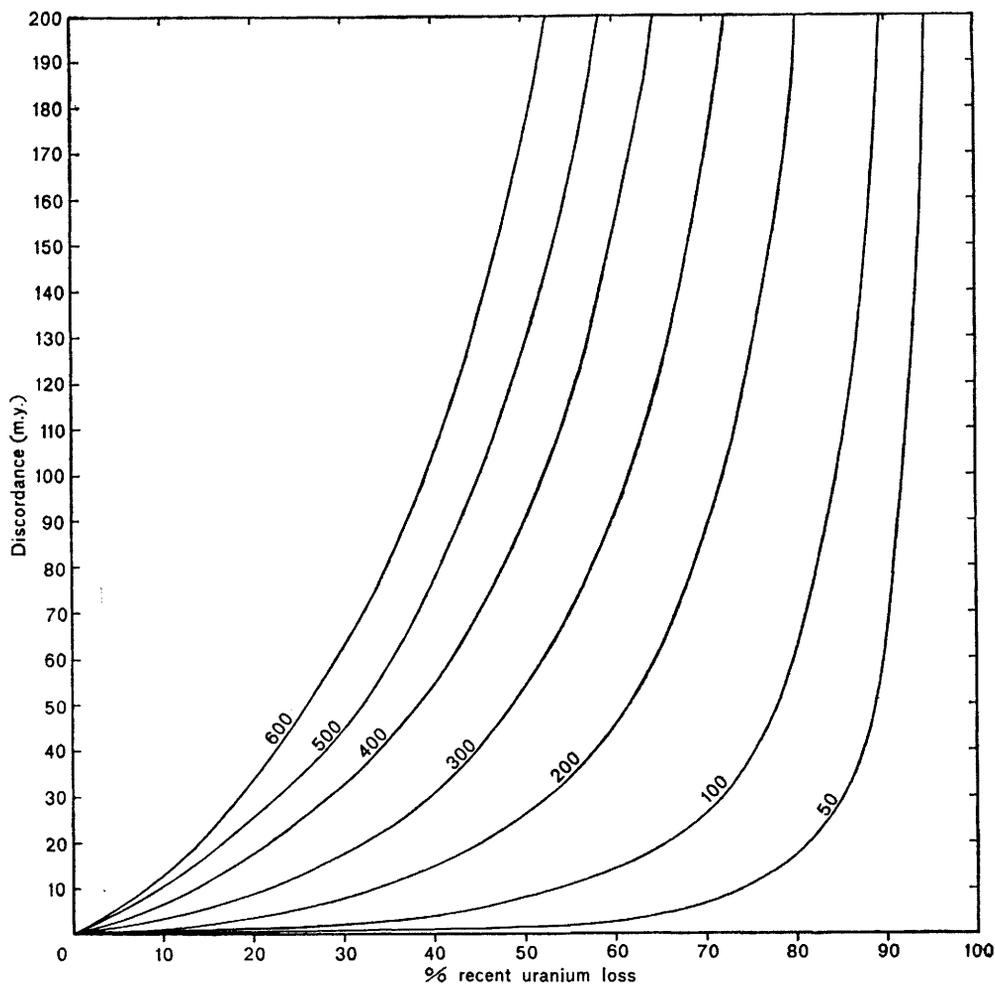


FIG. 5. Graph showing how the discordance between N_{206}/N_{238} and N_{207}/N_{235} apparent ages varies with increasing recent uranium loss for material of true age 50, 100, 200, 300, 400, 500, and 600 m.y.

of radon are readily detectable and it is quite feasible to measure the rate of radon loss from a specimen, so that the theory of radon loss can be tested.

In view of the limitations and ambiguities of interpretation presented by discordant ages, it would seem prudent to exclude any such results from serious consideration with respect to the Phanerozoic time-scale unless they can be taken in conjunction with results obtained by other methods, or unless arguments for accepting the value of one particular ratio are exceptionally well supported by independent evidence. For example, it is not sufficient to postulate that a certain result has been affected by recent lead loss unless this can be substantiated by detailed examination of the specimen material and its original environment. As

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far as possible uranium minerals suspected as having been altered by supergene action should not be selected for analysis.

6. The application of the uranium–thorium–lead method

The application of any age-determination method rests upon the availability of an analytical technique suitable for the material in question, which may be of very low grade, or very small in amount, or both. Apart from the lead-alpha method of dating zircons, most of the age-determination analyses of uranium and thorium minerals undertaken outside North America have entailed the determination of U, Th, and Pb by conventional chemical techniques. These suffer from the limitation that relatively large quantities of material are necessary if reproducible results are to be obtained. Further, if a mass spectrometer cannot be specifically reserved for geological work relatively large amounts of parent material are necessary. Under these circumstances it is usual to calculate the weight of material required for analysis on the basis of providing several milligrammes of lead. This facilitates the transfer of samples from one laboratory to another, their storage, and repeat runs in an instrument where background may be high owing to the variety of conditions under which it is used. Although the amounts required for U–Th–Pb analysis have not been large by comparison with those required for other techniques, the comparative scarcity of uranium and thorium minerals has frequently restricted application of the method in the past (see Table 1).

TABLE 1: *Approximate weight (in grammes) of material recommended for age-determinations where isotope dilution is not used*

| Age (m.y.) | 100 | 600 | 1200 |
|------------|-----|-----|------|
| Uraninite | 1 | 0.2 | 0.1 |
| Monazite | 25 | 5 | 2.5 |

However, the application of the isotope-dilution techniques of analysis to uranium first attempted by Tilton and his co-workers in about 1955 (see Aldrich [1956] for a review of this technique) has greatly increased the number of potential applications of the method, for it has improved sensitivity a thousand-fold. It is now feasible to attempt the analysis of a sample containing only 50 μg of uranium or lead, thus enabling zircon concentrates totalling only 0.2 g to be effectively analysed. Not only are the initial concentrates small in amount but the amount introduced into the mass spectrometer is correspondingly smaller than with earlier practice. Contamination must be rigorously avoided at all stages of the work and a specially designed laboratory and specially purified reagents are essential. Since the radioactive decay of uranium and thorium generates helium (see above), the measurement of U/He or Th/He ratios should provide a measure of age. The application of an isotope-dilution technique to the determination of uranium and helium according to recent work by Damon & Green (1963) has given grounds for believing that this method can be usefully reapplied. It was first attempted about

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1908 by Strutt, but fell into disrepute principally because of the poor helium retentivity of minerals with a high uranium content and the difficulties of measuring a very low uranium content. Using isotope dilution, Damon and Green have shown that non-metamict zircon and coarse (1 to 10mm) acid-washed magnetite can give ages consistent with those provided by other methods.

Uranium–thorium–lead age-determinations, like any other, can be undertaken only after suitable material has been found in the field. Such material is unlikely to be found without a careful study of the radioactivity, mineralogy, and geochemistry of any potential host rock. This necessitates the careful examination of such rocks in the field, using a sensitive ratemeter, and subsequent laboratory study to identify the source of the radioactivity, the nature of its environment, and its degree of alteration, if any.

The types of geological situation in which it is possible to find uranium or thorium suitable for stratigraphical dating can be summarized as follows:

(1) Igneous bodies that have a cross-cutting or uncomformable relationship with stratigraphically defined sediments. Accessory minerals in intrusions, pegmatites, or veins, or metasomatically formed minerals in contact zones can provide suitable material. Any non-metamict uranium- or thorium-bearing mineral may be used, as for example uraninite, pitchblende, monazite, zircon, or thorianite. In general, metamict minerals are not satisfactory, because their glassy structure readily permits the migration of elements, making them very susceptible to alteration. Nevertheless, useful results have been obtained, as for example from samarskite, brannerite, and davidite (Darnley *et al.* 1961). Individual occurrences of metamict minerals require particularly detailed laboratory assessment before age-determination analysis can be justified. There is renewed interest in the helium method, and magnetite in basic igneous rocks and contact zones may consequently become increasingly important in the future.

(2) Lavas or tuffs interbedded with stratigraphically defined sediments probably provide the most valuable material for defining points in the time-scale. Zircon is the most widespread constituent likely to be found in such rocks. Magnetite might be useful in the future.

(3) Sedimentary rocks. Uranium is readily adsorbed from sea-water by humic organic matter, certain clay minerals, and phosphates. As these may all be constituents of shale it is not surprising that the average uranium content of shales exceeds that of any other major group of sedimentary or igneous rocks, including granite. The best-known example of a shale with above-average enrichment is the Upper Cambrian Alum Shale of Southern Sweden. Cobb & Kulp (1961) carried out a detailed laboratory investigation and showed that the whole-rock specimens they examined gave discordant ages owing to removal of radiogenic lead which was enriched in ^{206}Pb as a probable consequence of ^{222}Rn diffusion. However, it seems possible that improved results might be obtained if specimens could be obtained from localities where the permeation of ground-water has been negligible. A more extended investigation than any so far attempted seems to be required to assess the application of whole-rock analysis to unmetamorphosed sediments.

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Phosphatic material can also be concentrated in certain limestones, especially those associated with reef-knolls. In the Lower Carboniferous of Derbyshire phosphates absorbed uranium, which is now present as uraniferous apatite (Peacock & Taylor, in the press). This might be suitable for dating, but since the uranium content is only about 0.1 per cent it would be necessary to use an isotope-dilution technique to carry out the analysis.

Thorium is precipitated under sedimentary conditions with hydroxides of iron and manganese, and as such may accumulate in shales. Little use has so far been made of syngenetic thorium in shale for dating purposes, although if absence of detrital thorium minerals could be reliably established the method deserves consideration.

(4) Heavy minerals in sediments. Dating of heavy minerals can provide only a maximum age, which might be useful in a region where deposition took place during active uplift. Uranium minerals formed at low temperatures do not survive transport, but thorium-bearing uraninite may do so in favourable circumstances, as in present-day River Indus sediments. Any non-metamict radioactive mineral present in sediments can be considered as potentially suitable for age-determination.

For the reasons outlined in an earlier section the uncertainty surrounding common lead ages is such that although they are of great value in the study of ore deposits and petrogenesis, they are at present of no practical value with respect to the time-scale.

It must be stressed that a good analysis is not in itself sufficient to guarantee the significance of a result. The whole environment of material selected for analysis must be carefully studied both in the field and in the laboratory and all aspects of field-relationships, mineralogy, and geochemistry, together with age-determinations by other methods, must be considered. No age-determination can be properly evaluated other than in its geological context.

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