DISCORDANT U-PB AGES AND MINERAL TYPE*

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ABSTRACT

The discordant ages obtained from the isotopic ratios Pb^{206}/U^{238}, Pb^{207}/U^{235}, Pb^{207}/Pb^{206} and Pb^{208}/U^{232} in various minerals are considered in terms of the mineral type. The available pertinent data including some new measurements made at this laboratory are compared. Each mineral structure appears to show a consistent but characteristic discordance, except monazite, which exhibits a dual behavior; i.e., pitchblende $206/238 < 207/235 <$ probable age; uraninite $208/232 < 206/238 < 207/235 <$ probable age; monazite, xenotime $208/232 <$ probable age $< 207/235 < 206/238$; monazite $208/232 < 206/238 < 207/235 <$ probable age; zircon and sphene $208/232 \ll 206/238 < 207/235 <$ probable age; samarskite, thorite, sphene $208/232 < 206/238 < 207/235 <$ probable age; euxenite $206/238 < 207/235 < 208/232$ = probable age. On the other hand if adequately preserved, concordant ages can be obtained from almost any radioactive mineral. Mineral structures differ considerably in their stability to alteration. For a given mineral the isotopes of lead derived from uranium decay must be in a different lattice environment than the Pb^{208} derived from thorium decay.

INTRODUCTION

The lack of agreement among the ages derived from the isotopic ratios of uranium, thorium and lead is one of the most important problems in geochronometry. Nier (1939) pointed out the problem in discussing his first isotopic ages by the uranium-lead method. Later, Holmes (1948) attempted to explain the most common age discordance of $Pb^{206}/U^{238} < Pb^{207}/Pb^{206}$ as a result of radon leakage, but Giletti and Kulp (1954) measured a representative suite of uranium minerals and found that the radon leakage for fresh primary minerals seldom exceeded a few per cent although in the case of very fine pitchblende, the value could reach 10% and some secondary minerals such as carnotite could reach 20–30%. If these data are combined with the observation of G. Tilton (personal communication) that the leakage under laboratory conditions will be maximal, it is concluded that radon leakage is not very important. For post-Cambrian minerals, however, the 207/206 age is quite sensitive to radon leakage.

Eckelmann and Kulp (1956) made a detailed study of the Lake Athabasca district pitchblendes. The ages within a specific sample as well as the ages from sample to sample showed major discordance. It was concluded that the dominant factor causing this discordance was lead loss and that the lead removal was most probably due to temperature rises at certain periods during the history of the mineral. This same pattern of ages appears at the Sunshine Mine, Idaho, and in the Witwatersrand

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pitchblende and thucolite deposits but is not evident at Katanga, Belgian Congo.

Stieff et al. (1953) have suggested that the deposition of old radiogenic lead along with the pitchblende can also explain the observed discordance and they cite evidence for this in the Colorado Plateau ores. Such a phenomenon could produce the observed isotopic ratios but would require the same hydrothermal solution which transported the uranium to also carry old radiogenic lead with essentially no contamination from common lead, and that deposition of the uranium and lead took place at the identical spots. It would be easiest for such a process to produce erroneous isotopic ratios in very young ores such as occur in the Colorado Plateau. Nevertheless if this were the case it would be expected that a representative sampling of intimately associated galena would show a preponderance of old radiogenic lead. Since this is not the case and since the observed pattern of ages is very slightly affected even if the most radiogenic lead is used for the corrections, the hypothesis of old radiogenic lead addition remains to be demonstrated. Certainly this process would not appear to be of any significance in pegmatite minerals.

Earlier some of the discordance of the ages was attributed to uncertainties in certain of the physical constants, particularly the half-lives of Th^{232} and U^{235}. The agreement in all of the isotopic ages for a few older samples (e.g. Bikita at 2650 m.y.) and the great variation in the degree of discordance observed with different minerals eliminate this as a significant source of error. Both the U^{235} and Th^{232} half-lives appear to be correct to at least 2%. Analytical error in the wet chemical method for lead can account for some cases of discordance in the analysis of lead in young minerals where the lead concentration is only a few tenths of a per cent. Here the apparent reproducibility is ±5% but the real value can be off by as much as 20% in some cases. This problem can be eliminated by using isotope dilution techniques.

Tilton (1956), who has conducted leaching experiments with dilute HCl on powdered samples for which the isotopic ages had been obtained, has examined the isotopic composition of the leach. He was able to show (1) in general, there is a correlation between the isotopic composition of the lead removed by dilute acid treatment and the observed age pattern obtained from the bulk sample (Tory Hill, Ont.). Thus in a zircon for which the Pb^{208}/U^{232} age is much lower than the Pb^{206}/U^{238}, the Pb^{208} is leached out more easily than Pb^{206} and Pb^{207}. (2) Thorium and uranium are removed in the leaching process as well as lead. In those samples where $206/238 > 207/206$, uranium is leached preferentially to lead where the $Pb^{208}/Th^{232} < Pb207/206$, Pb^{208} is leached preferentially to the other lead isotopes as well as to the thorium. (3) In the case of the samples that were analyzed, it was demonstrated that the readily leachable material was not added from outside since adjacent minerals give a totally different leach composition that is consistent with their own bulk composition. (4) The loss of lead isotopes during the history of a mineral is not necessarily proportional to the present abundance. Tilton's study did not represent the condition under which alteration of radioactive minerals actually takes place but clearly demonstrated that the readily mobile constituents in a given mineral structure are related to the age abundance. Ahrens (1955) has suggested that the high 206/238 ages for the Huron Claim and Las Vegas monazites are due to the error in the uranium analyses. It is not likely that this is the case, however, since the Carnegie Institute Annual Report 1954–1955 reports an identical pattern on analyzing monazite samples by the isotope dilution technique.

DISCORDANCE AND MINERAL TYPE

Enough data have now accumulated to warrant a preliminary analysis of the effect of mineral structure on the discordance of the ages. It appears from the foregoing discussion that loss of uranium, thorium and lead can take place under varying conditions. In a strongly acid environment such as might occur in the surface weathering of a pitchblende-sulfide complex, Phair and Levine (1952) have shown that uranium is leached preferentially to lead, but this situation is not a common one in nature. Føyn (1938) has reported an experiment in which lead exsolved from uraninite at elevated temperature (~300°) and pressure in a bomb with only water added.

The present isotopic composition in any sample, therefore, is dependent on the pressure, temperature and chemical environment to which a particular mineral lattice has been subjected at different periods in its history as well as the initial crystal structure.

Representative isotopic ages are compiled in Table 1 according to mineral type. The following conclusions can be derived from these data:

(1) Apparently all minerals can give concordant ages, i.e., Katanga pitchblende; Romteland, Norway and Nicolls Nob, Australia uraninites; Bikita, South Rhodesia and Goodhouse, South Africa monazites; Ceylon zircon and Marievale and Blijvoorvitzicht, South Africa thucolites. (It is interesting to note that the South African thucolites give concordant ages but apparently do not represent the true age of initial uranium mineralization. This is probably a case of "accidental concordance" described by Wetherill (1956).)

Examples of other minerals with concordant ages could doubtless be found if enough samples were analyzed. This observation shows that the

TABLE 1. ISOTOPIC AGES FROM VARIOUS URANIUM AND THORIUM MINERALS
(Age in m.y.)

A. *Pitchblende*

Locality	206/238	207/235	207/206	210/206	Reference
Colorado Front Range					
Wood Mine	55±1	56±2	150±110	—	Nier et al. (1941).
Richards Mine	56±1	62±6	320±230	—	Eckelmann & Kulp (1957).
German Mine	51±1	54±2	315±100	—	Eckelmann & Kulp (1957).
Sunshine Mine	805±10	860±20	1035±35	—	Eckelmann & Kulp (1957).
Great Bear Lake					
Eldorado Mine	1230±10	1305±15	1405±45	1230±40	Eckelmann & Kulp (1957).
Eldorado Mine	1010±5	1100±10	1240±20	1040±40	Eckelmann & Kulp (1957).
Eldorado Mine	1220±10	1275±15	1395±20	—	Nier (1939).
Saskatchewan-Manitoba					
(Lake Athabasca District)					
ABC Adit	335±15	395±15	730±30	355±8	Eckelmann & Kulp (1956).
Eagle Mine	895±5	975±10	1140±30	890±25	Eckelmann & Kulp (1956).
Ace Mine	1280±10	1490±10	1780±20	1280±30	Eckelmann & Kulp (1956).
Ace Mine	1550±10	1630±10	1795±15	1535±45	Eckelmann & Kulp (1956).
Belgian Congo, Africa					
Katanga	575±5	595±5	630±40	—	Eckelmann & Kulp (1956).
Katanga	616	614	610	—	Nier (1939).
Czechoslovakia					
Joachimsthal	178±2	195±10	385±135	—	Lamont (unpublished)
South Africa					
Witwatersrand	1380	1610	2070	—	Lamont (unpublished)

B. *Uraninite*

Locality	206/238	207/235	207/206	208/232	Reference
Huron Claim S.E. Manitoba	1564	1985	2475	1273	Nier (1939).
Huron Claim S.E. Manitoba	1860	2170	2505	1360	Eckelmann & Kulp (1957).
Viking Lake Manitoba	1850	1880	1920	1670	Wasserburg & Hayden (1955).
Viking Lake Manitoba	1525	1720	1870	445	Eckelmann & Kulp (1957).
Lac La Ronge Manitoba	565	868	1750	—	Eckelmann & Kulp (1957).
Black Hills South Dakota	1580	1600	1630	1440	Tilton et al. (1956).
Black Hills South Dakota	1615	1615	1622	1370	Eckelmann & Kulp (1957).
Black Hills South Dakota	1390	1460	1610	1030	Eckelmann & Kulp (1957).
Wilberforce	1077	1050	1035	983	Nier (1939).
Wilberforce	1015	1038	1060	—	Eckelmann & Kulp (1957).
Wilberforce	1000	1015	1030	1010	Nier (1941).
Wilberforce	1000	1020	1085	870	Wasserberg & Hayden (1944).
Wilberforce	994	993	982	897	Wasserberg & Hayden (1955).
Romteland Norway	890	892	920	900	Lamont (unpublished).
Nicoll's Nob Australia	445±5	430±15	340±75	435±15	Lamont (unpublished).

Table 1—*Continued*

Locality and Mineral	206/238	207/235	207/206	208/232	Reference
C. *Monazite*					
Ebonite Claims So. Rhodesia	2640	2670	2700	2640	Holmes (1954)
Jack Tin Claims So. Rhodesia	2210	2460	2660	1940	Holmes (1954)
Irumi Hills No. Rhodesia	1990	2330	2640	1380	Holmes (1954)
Huron Claim Manitoba	3220	2840	2590	1830	Nier (1939)
Antsirabe Madagascar	1370	1850	2450	610	Holmes and Cohen (1955)
Yadiur India	1410	1820	2330	1800	Holmes (1955)
Ene India	4300	3100	1600	540	Holmes (1955)
Las Vegas New Mexico	1730	1560	1340	770	Nier et al. (1941)
Quartz Creek Colorado	1590	1420	1170	995	Tilton (1956)
Mount Isa Australia	—	—	1160	1000	Nier et al. (1941)
Steenkampskraal So. Africa	1080	—	—	990	Tilton and Nicolaysen (1957)
Soniana India	635	700	910	610	Holmes et al. (1949)
Houtenbeck So. Africa	1400	1230	930	940	Tilton (1956)
Goodhouse So. Africa	930	915	880	900	Tilton (1956)
D. *Zircon*					
Quartz Creek Colo.	930	1130	1540	515	Carnegie Re. 1954–1955.
Tory Hill, Ont. (−200 mesh)	1030	1055	1090	390	Carnegie Re. 1954–1955.
Tory Hill, Ont. (1–2 mm)	940	960	1015	—	Carnegie Re. 1954–1955.
Bagdad, Ariz. (−200 mesh)	630	770	1210	270	Carnegie Re. 1954–1955.
Natural Bridge, N.Y. (2–3 cm)	1025	1065	1140	—	Carnegie Re. 1954–1955.
Ceylon (2–3 cm)	540	544	555	538	Carnegie Re. 1954–1955.
Cape Granite South Africa	330	355	525	237	Carnegie Re. 1954–1955.
E. *Samarskite*					
Petaca District New Mexico	240	310	875	180	Eckelmann & Kulp (1957).
Spruce Pine North Carolina	282	292	405	170	Eckelmann & Kulp (1957)
Spruce Pine North Carolina	307	312	380	205	Eckelmann & Kulp (1957).
Spruce Pine North Carolina	314	316	342	302	Eckelmann & Kulp (1957).
F. *Euxenite*					
Romteland Norway	485	560	910	920	Lamont (unpublished).
Kalstad Norway	450	530	940	980	Lamont (unpublished).

TABLE 1—*Continued*

Locality	206/238	207/235	207/206	208/232	Reference
			G. *Microlite*		
Harding Mine New Mexico	916	996	1180	—	Carnegie Inst. Re. 1954–1955.
Quartz Creek Colorado	915	1055	1350	—	Tilton (1956).
			H. *Others*		
Romteland, Norway (Thorite)	740	760	830	594	Lamont (unpublished).
Swaziland, Africa (Yttrotantalite)	1105	1455	2060	1270	Lamont (unpublished).
Quartz Creek, Colo. (Columbite-tantalite)	1520	1470	1390	—	Tilton (1956).
Uncompahgre, Colo. (Biotite-xenotime)	3180	2065	1640	1100	Tilton (1956).
Tory Hill, Ont. (Sphene)	910	—	—	—	Tilton (1956).
Tory Hill, Ont. (Sphene)	—	—	1090	450	Tilton (1956).
Besner, Ont. (Thucolite)	262	275	440	250	Nier (1939).
Witwatersrand (Thucolite)					
Black Reef	2110	2310	2510	—	Louw (1955).
Western Reefs	1690	2010	2390	—	Louw (1955).
Marievale	2220	2170	2100	—	Louw (1955).
Blijvoorvitzicht	2110	2080	2070	—	Louw (1955).
Babrasca	1910	2060	2250	—	Louw (1955).

alteration involving the differential loss of lead, uranium and thorium is not a normal continuous process throughout the life of a mineral. Even though a mineral specimen is structurally capable of differential loss of lead, uranium or thorium, it will not undergo alteration unless the proper external conditions are existent.

(2) The degree of discordance is essentially independent of the age of the mineral. The variation in the 207/206 ages from different pitchblende samples from the same deposit proves that the major alteration did not occur recently. Therefore this discordance can not be attributed to supergene effects. Where a sample is weathered as in the case of the Katanga curite analyzed by Nier (1941), the age pattern can be reversed. For this to occur the mineral usually exhibits secondary uranium minerals.

(3) The fact that the 207/206 ratios of the radiogenic component of the lead isotopes in galena and clausthalite associated with pitchblende are closely grouped suggests that lead loss occurred during certain discrete events or intervals (Eckelmann and Kulp, 1956) and was not a continuous process. Temperature changes have been suggested as the most likely cause (Eckelmann and Kulp, 1956) but certain chemical environments may be required.

(4) Uraninite shows the age pattern $Pb^{206}/U^{238} < Pb^{207}/U^{235} < Pb^{207}$

$/Pb^{206}$. It also shows the apparent age relation of $Pb^{208}/Th^{232} < Pb^{206}/U^{238}$. Even if the uranium lead ages are concordant within the experimental errors, as is the case with the Viking Lake and the two Black Hills specimens, the Pb^{208}/Th^{232} ages show evidence of lead loss. Therefore the Pb^{208} is in a different structural environment than the Pb^{206} and Pb^{207}. The Wilberforce samples show a discordance slightly outside the experimental error but the age pattern is consistent with other uraninites. The ages for the one Wilberforce sample analyzed by Nier (1941) which show $Pb^{206}/U^{238} > Pb^{207}/Pb^{206}$ are actually within the experimental errors and the particular pattern is possibly the result of a slight error in the lead analysis. If the lowest lead analysis reported for this sample is used the Pb^{208}/Th^{232} age is lowered by about 40 m.y. The Wilberforce uraninite age pattern does not preclude the alteration having been fairly recent but more data may alter this conclusion. If the 207/206 ages of 1085 m.y. and 1012 m.y. are outside of the experimental error, this is evidence for lead loss in the past.

(5) The 208/232 age is lower than the 207/206 age in proportion as the 206/238 age is lower than the 207/206 age. This is shown best by the Huron Claim and Viking Lake samples. The Nicoll's Nob, Romteland and several Wilberforce samples are essentially concordant.

(6) Monazite shows two major types of discordance, i.e., $206/238 > 207/235 > 207/206 > 208/232$ and $207/206 > 207/235 > 206/238 > 208/232$. The particular conditions under which one or the other of these effects may be produced are not yet defined, but the alluvial monazite from Ene, India shows the greatest discordance suggesting that the Pb^{208} and uranium loss required for this discordance may be supergene, whereas the other pattern may develop under conditions of elevated temperature. It is clear from the monazite data on localities dated by other methods that the Pb^{208} is easily mobilized and that the uranium and its associated lead isotopes are removed in different relative amounts depending on the conditions. In most cases it would appear that the 207/206 age is closest to the true age. If the 207/206 and 208/232 ages agree this should be good evidence of the true age. In the case of the Goodhouse monazite, which yields concordant ages, acid leaching experiments by Tilton (1956) removed ratios of the uranium, thorium, and lead similar to those existing in the bulk mineral. Furthermore the sample was very homogeneous upon microscopic examination in contrast to the Quartz Creek monazite, which showed extensive orange-brown fillings in fractures.

Rubidium-strontium ages on the Southern Rhodesia localities reported by Aldrich, Wetherill, et al. (1956) and Huron Claim samples at Lamont show that in these cases of considerable alteration the 207/206 age is correct within the experimental error. The Quartz Creek, Colorado locality, however, appears to have a true age of 1350 ± 100 million years (Aldrich, Davis, et al. 1956) so that the 207/206 age of 1170 ± 50 is somewhat low indicative of lead loss early in the history of the mineral.

(7) Zircon shows a similar age pattern to uraninite, i.e. $Pb^{208}/Th^{232} < Pb^{206}/U^{238} < Pb^{207}/U^{235} < Pb^{207}/Pb^{206}$ except that the Pb^{208}/Th^{232} ag is even more sensitive to alteration. In this regard it is noteworthy that Tilton (1956) found a Pb^{208}/Pb^{206} ratio ten times larger in this acid wash than in the bulk Tory Hill zircon. Concordant results are obtained in the case of the excellent, large Ceylon crystals. The size of the crystal appears important. Zircons present as fine accessory minerals in a rock are more subject to alteration than coarse crystals. All of these samples were fresh and showed no evidence of surface weathering. Moreover most of the Pb^{208} at Wilberforce must have been lost subsequent to 390 m.y. ago. There appears to be no clear relationship between the extent of discordance and age. It would be of interest to analyze zircon from Bikita, So. Rhodesia to check this point.

(8) Samarskite shows a similar pattern to zircon. It should be noted these these samples were clean and glassy without a trace of alteration. The true age of the Spruce Pine samples is probably close to 350 m.y.

(9) Euxenite has a characteristic age pattern showing the Pb^{206} and Pb^{207} to be much more sensitive to removal than the Pb^{208} so that, at least for mild conditions, Pb^{207}/Pb^{206} age $= Pb^{208}/Th^{232}$ age which are probably close to the correct age.

(10) Microlite shows the apparent age relationships $Pb^{206}/U^{238} < Pb^{207}/U^{235} < Pb^{207}/Pb^{206}$. If the Harding Mine microlite and Petaca samarskite samples are actually the same age the greater sensitivity of samarskite to lead loss is indicated.

(11) Thorite, sphene and thucolite show a zircon-like pattern.

(12) Columbite-tantalite and xenotime show $Pb^{206}/U^{238} > Pb^{207}/U^{235} > Pb^{207}/Pb^{206}$ indicative of preferential uranium loss. The xenotime also shows evidence of Pb^{206} loss. Yttrotantalite is different from columbite in that the former shows $Pb^{206}/U^{238} < Pb^{207}/U^{235} < Pb^{207}/Pb^{206}$. It is different from the zircon and uraninite in that the uranium lead appears more readily leachable than the thorium lead.

Several localities for which more than one mineral has been analyzed are listed in Table 2. This compilation permits a comparison of the relative sensitivities to alteration under apparently identical conditions. The following conclusions appear warranted.

Under conditions of slight alteration e.g., Romteland, Norway, where the uraninite ages are essentially concordant, the uranium lead in euxenite is readily removed, but the thorium lead and lead ages are concurrent.

TABLE 2

Mineral	206/238	207/235	207/206	208/232	Reference
A. Romteland, Norway					
Uraninite	890±5	892±8	920±20	900±30	Lamont (unpublished).
Euxenite	485±5	560±7	910±20	920±35	Lamont (unpublished).
Thorite	740±6	760±8	830±25	495±30	Lamont (unpublished).
B. Huron Claim, Manitoba					
Uraninite	1564	1985	2475	1273	Nier (1941).
Uraninite	1860	2170	2505	1360	Lamont (unpublished).
Monazite	3217	2839	2590	1827	Nier (1941).
C. Quartz Creek, Colo.					
Monazite	1590	1420	1170	995	Tilton (1956).
Zircon	930	1130	1540	515	Carnegie Re. 1954–1955
Microlite	916	1055	1350	—	Tilton (1956).
Columbite-tantalite	1520	1470	1390	—	Tilton (1956).
D. Wilberforce Area					
Uraninite	1077	1050	1035	983	Nier (1939).
Uraninite	1015	1038	1060	—	Eckelmann & Kulp (1957).
Uraninite	1000	1015	1030	1010	Nier (1941).
Uraninite	1000	1020	1085	870	Wasserburg & Hayden (1955).
Uraninite	994	993	982	897	Wasserburg & Hayden (1955).
Zircon (1–2 mm.)	1030	1055	1090	390	Carnegie Re. 1954–1955.
Zircon (−200 mesh)	940	960	1015	—	Carnegie Re. 1954–1955.
Sphene	910	—	—	—	Carnegie Re. 1954–1955.
Sphene	—	—	1090	450	Tilton (1956).

This may be the explanation for the low chemical ages on euxenite in the Grenville Province reported by earlier workers. Thorite readily loses Pb^{208}. It is noteworthy that thorite is generally found outside of radioactive equilibrium (F. Senftle, private communication). The low Pb^{207}/Pb^{206} age for the Romteland thorite strongly suggests that the uranium lead removal occurred during a condition which did not affect the uraninite. This event or events could not have been recent but must have occurred in the geologic past.

The same mineral type in the same pegmatite may show quite different degrees of alteration, i.e. Huron Claim and Black Hills uraninites. Where uraninite is strongly discordant, monazite is also (Huron Claim). The Huron Claim data suggest a probable age for the deposit of at least 2600 m.y. The Pb^{207}/Pb^{206} ages from the uraninite reflect lead loss in the past. If uranium loss occurred in the monazite in the past, the 207/206 age would be too low.

Where other minerals such as uraninite (Huron Claim) and zircon (Quartz Creek) are strongly altered, monazite loses Pb^{208} as well as uranium.

Microlite has a stability similar to zircon under the same conditions.

The stability of zircon to alteration is similar to the stability of uraninite except that zircon can lose Pb^{208} more readily.

It is becoming clear that there are many factors which play a role in producing discordant uranium ages. Some of the effects can be predicted, measured and corrected, whereas others cannot be inferred quantitatively as yet. It appears that the Pb^{207}/Pb^{206} age is the best first approximation of the true age for all of these minerals and will almost always be minimal. The degree of uncertainty is related to the number of samples analyzed. If several samples of two or three mineral types can be analyzed from a given locality, it is possible to assign an age within fairly narrow limits. Thus despite discordance, the following ages are very likely: Romteland, Norway 920 ± 20 m.y.; Huron Claim, Manitoba 2600 ± 100 m.y.; Black Hills, South Dakota 1620 ± 20 m.y. and Wilberforce, Ontario, 1060 ± 20 m.y. Other age assignments can be made with a slightly larger error.

It remains to investigate the particular mechanism of removal in nature and to relate this quantitatively to the mineral structure.

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