INTRODUCTION

This paper describes a piece of research that Dr. Rado Gospodarič and I pursued together between 1978 and 1983. It is with great sadness that I write it and make the interpretations without him. Rado and I were good friends since we first met at the 5th International Speleological Congress, Stuttgart, in 1969. After that we were together in the field at different times in Slovenia, in the high Rocky Mountains of Canada and in Kentucky. In July 1987 Rado and his daughter were my guests in the dolomite island karst of Lake Huron; it was a brief visit before he went to the INQUA congress in Ottawa where he presented a preliminary paper that gave his interpretations of the speleothem dating work we had done together (Gospodarič, 1987). At that time we planned to conclude our analysis and write a joint paper during the early summer of 1988. It was not to be. Here I present my interpretation of the results – to commemorate a dear friend.

Rado's greatest interest, perhaps, was in sedimentary deposits in the Slovene caves. Many readers will appreciate that such deposits can be very complex, often bewildering, stratigraphic and sedimentological phenomena. This is because of the rapid lateral and vertical transitions that can occur in caves, and because there may be much reworking of older deposits or intrusion of newer ones into a stratigraphic section. Deposits often contain allochthonous detritus (sand, silt, clay, etc.), allochthonous and autochthonous organic remains (bones, nests) and autochthonous precipitates ("speleothems", such as calcite flowstones and stalagmites) that are interlayered in complex sequences. The differing materials represent the predominance of different processes at different times during the accumulation of these piles of sediments.

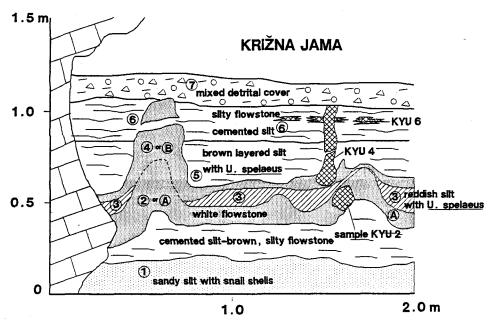
Many calcite speleothems can be dated by the Uranium series radiometric methods. This dating is, at the present time, the principal means of ordering and correlating cave sediments that are older than the (approximately) 38000 years BP limit of the carbon—dating method. However, difficult sedimentological and other problems can be encountered in U series dating, especially where the flowstone layers are mixed irregularly with clastic and organic deposits. Investigators should never be satisfied with just one or two U series dates from a complex stratigraphic section; there must be repeat measurements, plus new samples from lateral deposits that are believed to correlate with those already dated. It is best to return to the site after a first series of dates have been obtained, because those dates may suggest that the earlier stratigraphic mapping was in error.

The problems that may arise when attempting to date even quite simple sequences of deposits in caves are well illustrated by the project that Rado and I undertook together in the great cave, Križna Jama, near Postojna.

THE URSUS SPELAEUS DEPOSITS IN KRIŽNA JAMA

Križna Jama is a river cave that drains Bloke Polje into Cerknica Polje, Slovenia. The modern river passage is large and at 610 m above sea level. It is prone to violent flooding. There are older passages between 620 and 640 m that no longer flood; they contain major deposits of fluvial sediments and some speleothems. One of these passages, Medvedji rov (Bear Passage) has a big, protected recess where some silts containing large numbers of bones of *Ursus spelaeus* are interlayered with flowstones.

The apparent stratigraphy of the site is shown in Figure 1. Rado took me there in April 1978 as part of a general geomorphic excursion in the cave. At that time he supposed that the bones were of Early or Middle Wurm age; they were known to be older than 38000 years. Their close stratigraphic association with flowstones made it worthwhile to attempt U series dating. Small samples were taken from the flowstone layers immediately above and below the principal *Ursus spelaeus* remains at a point approximately two metres to the right of the sections illustrated in Figure 1.



Sl.1 Stratigrafija plasti jamskega medveda v Medvedjem rovu Križne jame, ki kaže lokacijo vzorcev kapnika KYU 2, 4 in 6. Meritev in risbo sta izdelala R.Gospodarič in M.Kranje decembra 1982.

Fig.1 The stratigraphy of the *Ursus spelaeus* deposits in Medvedji rov, Križna Jama, showing the locations of speleothem samples KYU 2, 4 and 6. Survey and drawing by R. Gospodarič and M. Kranjc, December 1982.

U SERIES DATING

The natural isotopes, 238 U, 235 U and 234 U occur in trace amounts in all rocks and soils and can be quite abundant in felsic volcanic rocks and black shales. Their radioactive decay by (4 He), and emissions to stable 207 Pb, 206 Pb offers many potential dating schemes. The most important is the decay of 234 U to 230 Th. When weathered from rocks, uranium is readily soluble in ionic forms such as $UO_2(CO_3)^{2+}$, and thus may be coprecipitated when calcite is precipitated from saturated ground water. The thorium or protactinium to which U decays is insoluble in normal waters and so is not precipitated in speleothem. If the relative abundance of 238 U, 234 U and 230 Th can be determined by sampling their respective particle production rates (alpha spectrometry) it is possible to obtain the age of the host sample in the range, 1000 to 350000 or 400000 years BP.

²³⁰Th: ²³⁴U dating by alpha spectrometry is now the standard method. 10 to 50 gms of calcite sample (depending upon the abundance of U and Th in it) are dissolved and the U and Th extracted or separated in a 20-step extraction process. These isotopes are then plated out onto separate stainless steel discs and placed in alpha counters until approximately 10000 decay counts have been recorded. There are many lengthy reviews giving details of procedures (e.g. Cherdyntsev,1971; Ivanovich and Harmon,1982; Ford and Schwarcz, 1981; Schwarcz,1980, for the particular problems of archaeological sites;etc.). The McMaster University laboratory directed by Professor Schwarcz and me since 1970 is one of the leading centres of speleothem dating.

Not all speleothems can be dated. Most reviews emphasize that there are three principal requirements for success:

- 1. There must be sufficient U in the sample. With modern extraction procedures it is possible to work with as little as 0.01-0.02 ppm U; however 1 or 2 ppm is preferred because the accuracy of the results is greatly improved.
- 2. The calcite cristal lattice (with its trace U content) must be impervious. If it is not, later groundwaters may leach some U atoms from it preferentially. This results in the calculation of too great an age for the sample.
- 3. There must be little or no thorium from particles of detritus trapped in the calcite as it is accumulating. Although thorium does not dissolve, it readily bonds to clay colloids or other solids in suspension in water. If the speleothem has a large content of detrital clay it is likely also to have a large content of detrital thorium; this results in the calculation of too great an age for the sample.

Detrital thorium can be a great problem. We attempt to estimate its significance by comparing the abundance of ²³⁰Th (which can derive from detritus or from decay of ²³⁴U) to that of ²³²Th (which can only derive from detritus). Where the ratio, ²³⁰Th: ²³²Th, is greater thus 20:1 it is safe to assume that detrital thorium is not significant.

In addition to these three standard requirements, our McMaster laboratory experience of the past 15 years suggests a fourth – that there be little or no organic matter contained in the

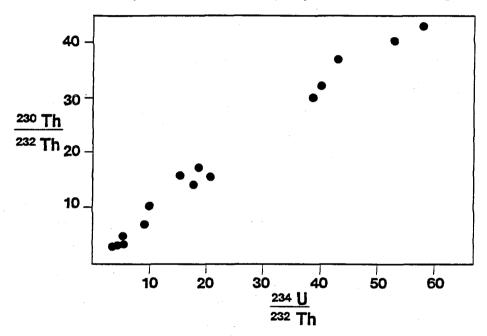
TABLE 1. U SERIES DATES FROM U. SPELAEUS SITE, KRIZMA JAMA.

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speleothem. Organics appear to chelate U and Th during the extraction processes, and may remove part or all of one or the other of them. This is not determined until counting is complete. Then it is learned that the entire preparation, extraction, plating and counting process (10 days' work) was wasted on the sample. It must be repeated or the sample abandoned.

RESULTS

Results of the analyses at McMaster University are presented in Table 1 and Figure 2.



Sl.2 Izohronska risba razmerij ²³⁰Th : ²³²Th in ²³⁴U : ²³²Th analiz, predstavljenih na tabeli 1 - podatki U serije z najdišča jamskega medveda v Križni jami. Nagib črte je izohronska starost približno 150.000 let BP.

Fig.2 Isochron plot of the ²³⁰Th: ²³²Th and ²³⁴U: ²³²Th ratios of the analyses cited in Table 1. U series dates from *U. spelaeus* site, Križna Jama. The slope of the line is an isochron age of approximately 150000 y B.P.

Specimens collected in April 1978

Rado and I collected specimens of 500 gms each from the flowstones above and below the principal *Ursus spelaeus* layer. The specimen above was given the code number, YUG K1, and is believed to be lateral equivalent of Layer 4 or B in the section given in Figure 1. The lower specimen was coded YUG K2 and is believed to be equivalent to Layer 2 or A. During 1980 and 1981 a total of nine samples were taken from these two specimens, consuming them completely. From the Table it is seen that only four samples gave publishable results. In the other five cases, U was largely or entirely lost during extraction.

From this first set of analyses it became apparent that all four of the problems listed above that can arise in U series studies are encountered at the Križna Jama site.

1. The U content of speleothems is very low (0.07 - 0.2 ppm only). The same is true of samples from Postojna Jama also; in fact, we are finding that low U is typical of holokarstic sites around the world (e.g. in Jamaica; Belize; at Guilin, China, etc.). Much better results are obtained at sites where shale caprocks are near (e.g. Kentucky; Yorkshire; Nahanni, Canada).

Because of the low U content, 35 to 50 g of calcite must be taken to obtain sufficient U and Th atoms for an analysis. This represents three or four cm³ of material. From sites that have flooded (such as Križna Jama) such large volumes of calcite will almost inevitably contain detrital clay with thorium, organic matter, and perhaps vuggy patches where U leaching can occur.

- 2. The 1978 samples do have a large content of detrital thorium. In Table 1 it is seen that three of the four "successful" results have ²³⁰Th: ²³²Th ratios far below the value of 20:1 that is considered to be the lower limit for clean, detritus—free, calcite. This is despite the fact that all samples were subject to careful cleaning procedures. Note that, where there is a high detrital content, it is the convention to calculate two ages: (i) a simple age assumes that the initial ²³⁰Th: ²³²Th ratio was 1.0 in the detritus, and (ii) a "corrected" age that assumes that the initial ratio was 1.25 or 1.50 (see Ku 1976 for details).
- 3. There appears to have been a lot of organic matter contained in the feed waters from which the calcites at the site were deposited. Even samples of white or translucent calcite from the 1982 collections yielded an organic foam upon dissolution in HCL. As a consequence U yields from the extractions were generally low, as will be seen if they are compared to Th yields throughout Table 1. Altogether, a total of 31 analyses were attempted from the three collections; they yielded only 16 publishable results, chiefly because U yields were too low or zero.

From the April 1978 collection only one analysis, YUG K1A Repeat, is of truly acceptable quality. This was an analysis of four cm³ of calcite with minor clay, from the lower half of the original specimen. It was ground and hand—screened before dissolution, when it appeared to be free of clay. Its date suggested that this flowstone layer began growing upon the principal *Ursus spelaeus* layer at some time between 135000 and 165000 years BP. Rado suggested that this might be too early for *Ursus spelaeus*. The calcite had contained vugs, so it was possible that preferential leaching of U had occurred after the flowstone was deposited; this would cause too great an age to be calculated. To resolve this problem Rado made a second collection at the site in April 1982.

Specimens collected in April 1982

Rado deliberately sought the largest volumes of the cleanest material available in the two flowstone layers. He found these several metres from our previous collecting position. They were two stalagmites growing in and on the flowstones. They were not emplaced directly above one another, but were offset approximately one metre apart and at the same elevation.

YUG K2R was a portion of a stalagmite that Rado belived to be from Layer 2 (Figure 1). It was tapered, and 11 - 14 cms in diameter. It consisted of clean, translucent to white calcite with a weathered outer surface. A dirtier, vuggy layer in the centre represented a possible hiatus in growth. Part of the true base of the deposit (silt cemented by calcite) had been removed before it was shipped to McMaster University.

Given the difficulties of low U content and complications from organic matter present, this specimen yielded excellent results. Analysis K2R Base was of the basal one cm of clean calcite. K2R Middle was a one cm thick layer of darker calcite 5 cm above the Base sample, and just above the central vuggy layer. K2R Top was the top one cm after removal of the weathered, outermost three mm. It was 1.5 to 2.5 cm above the Middle sample.

From the dates, it appears that this specimen grew rather steadily between approximately 146000 and 126000 years BP, when it was abruptly buried by deep silt containing *Ursus spelaeus* bones. There was a brief cessation of deposition (possibly caused by a minor flood) shortly before the Middle date of 132000 years. The rate of extension of this stalagmite averaged 0.25 cm per thousand years.

Specimen YUG K4 was a section across the base of a stalagmite 9 to 15 cm in radius. Rado believed it to be from Layer 4 in Figure 1. Its core had high vug porosity, with intruded clay. The outer growth was of finely laminated, white calcite; there was one minor but distinct hiatus in growth halfway through it.

Analysis YUG K4 Base was of ground calcite from the vuggy core that was washed, hand-screened, and leached to 50% dimensions in HCL before standard dissolution and extraction. Its very low U content and its great age suggest that despite these precautions, the analyzed sample had suffered preferential leaching of U.

Analysis YUG K4 Middle was of a one—cm layer of the laminated calcite directly below the central hiatus. It indicates that growth halted for an unknown (but probably short) time interval around 170000 +- 25000 years BP.

The top date was of calcite 0.8 to 2.0 cm above the hiatus; an outermost 2 mm of weathered material was removed. A problem with an alpha particle counter compelled us to reduce the counting time for the Thorium spectrum of this sample to well below the norm. This explains the very wide error margin (one standard deviation) about the calculated age of 146000 years. There were two further attempts to date this layer; U was lost in one case, and Th in the other.

Specimen YUG K4 began to grow at some unknown time before 170000 y BP. Growth halted for an unknown duration. Thus, the hiatus may coincide with the coldest period of the penultimate glaciation. Growth was renewed, and then terminated when the deposit was buried by silt containing *Ursus spelaeus* remains at 146000, + 44000, and – 31000 years BP.

The six dates from two flowstones buried by *Uspelaeus* silts give a perfect stratigraphic order. This is rare in U series dating by alpha spectrometry, and perhaps unprecedented in deposits with low U and high detrital Th problems such as are encountered at this site. Therefore, they appear to be a most satisfactory set of results. However, the great problem is that the younger stalagmite from the U series results, K2R, was assigned by Rado to the older stratigraphic position (Layer 2), and vice versa. Either there had been a mis—assignment at this complex site, or a mistake in labelling afterwards. Because of this and because of the poor U yields attributable to problems with organics (especially for the analyses, K4 Top, K2R Base and K2R Middle that lie around what appears to be a critical date of 146000 years), Rado decided that he must take a third collection.

Specimens collected in December 1982

Rado was assisted by Mrs. Maja Kranjc. The site was measured carefully for the preparation of Figure 1. Three new specimens were taken, as shown in the figure. These were given the general designation, KYU, in order to avoid confusion with the two earlier YUGK collections. KYU 2 was from Layer 2 or A; KYU 4 was a candlestick stalagmite growing from the Layer 4 (or B) flowstone and truncated by erosion at the Layer 6 – to – 7 contact; KYU 6 was a calcitic overgrowth on this stalagmite that was deposited and then buried during the accumulation of the Layer 6 cemented silts.

The data shown in Table 1 tend to uphold the old scientific notion that when a good result has been obtained (as it was from the April 1982 collection) the workers should halt the research!

Specimen KYU 2 was a flowstone 8 cm thick. It has a dirty base with high vug porosity, plus minor hiatuses with clay and vugs at 4 cm and 6 cm above the base. A total of eight analyses were attempted, all Th being lost in two, and most U in two others. Analysis KYU 2 Base was of 2 cm of discoloured calcite immediately above the dirtiest, basal material. Its date is broadly concordant with the YUG K4 Base and K4 Middle results. Analysis KYU 2 Middle incorporated clay or vuggy calcite from the + 4 cm hiatus; evidently, the attempts to clean it failed because it yielded an indeterminate age. Analysis KYU 2H was of a one cm layer of cleaner calcite between the +4 and +6 cm hiatuses; it, too, is broadly concordant with the YUG K4 Middle date. Analysis KYU 2 Top was 1 to 2 cm above the +6 cm hiatus; the calcite was vuggy and contained intruded silt which, clearly, created analytical difficulties.

The candlestick stalagmite, KYU 4, was 3.5 cm in diameter. Upon sectioning it was found to be dirty and with many vugs troughout its length. Four out of five analyses failed due to loss of most or all U or Th. Analysis KYU 45 Base was prepared with great care and all cleaning techniques applied; its thorium yield can be judged a failure, but it gives an age that is broadly concordant.

Specimen KYU 6 was a mixture of layered silt cemented by calcite and layered calcite impregnated with silt. There is too much detrital Th contamination for significant results; one analysis that yielded a finite age is cited in Table 1 as an example.

DISCUSSION AND CONCLUSION

Isochron age

It has been emphasized that, because of their low U content, high detrital and organic contamination and possible leaching, the Križna Jama speleothem deposits at the *Ursus spelaeus* site present great difficulties for conventional U series analysis by alpha spectrometry. Such intractable deposits are often encountered at archaeological sites in cave entrances and on spring travertines. For such deposits, Schwarcz (1980) has proposed an isochron dating technique where, if repeated analyses of a very dirty specimen yield a good linear relationship in the ratios, ²³⁰Th: ²³²Th v ²³²U: ²³⁴Th, the slope of the line is indicative of the true age of the deposit. Figure 2 is such an isochron plot for all of the samples listed in Table 1. It is seen that there is a good linear relationship. The slope of the line yields an isochron mean age of 150000 years BP for the entire collection.

The nature and age of Ursus Spelaeus Layer 3

It is my opinion that the stratigraphic sequence of dates obtained from the April 1982 collection of speleothems should be accepted as it is presented in Table 1. YUG 4K represents flowstone of Layer 2. YUG K2R represents flowstone of Layer 4. There was a labelling accident or mis—assignment at the site.

From that sequence of dates and the site sedimentology the following record appears:-

- 1. Growth of flowstone Layer 2 was terminated around 146000 BP by the aqueous or colluvial deposition of silts containing bones and bone fragments of *Ursus spelaeus*.
- 2. The duration of silt Layer 3 deposition was too short to be resolved by the U series dates. It was possibly a single flood event of exceptional magnitude. It is the brevity of this event that contributes to the difficulty in distinguishing flowstones that grew before and after it.
- 3. Deposition of flowstone Layer 4 began on top of this silt around 146000 years and continued until 126000 years BP. Then it also was terminated by rapid burial in silt containing bones of *Ursus spelaeus*, = silt Layer 5.
- 4. Calcite deposition resumed during Layer 6, but there was now a steady supply of airborne or waterborne silt to contaminate it.
- 5. The top of the section (Layer 6) was truncated by flood action. The mixed clastic deposits of Layer 7 have accumulated upon the truncation surface since that event.

The April 1982 U series age estimate of approximately 146000 years for the Layer 3 event is strongly supported by the isochron result, and by the U series ages for YUG K1A Repeat, YUG K2A Repeat ("corrected" age), and KYU 4S Base. YUG K4 Middle, KYU 2 Base and KYU 2H are acceptable that confirm that flowstone Layer 2 was growing, with one or two hiatuses, for 20000 to 40000 years before deposition of Layer 3 terminated it. The YUG K2R Middle and Top dates show us that the same mode of calcite deposition resumed at the site after the Layer 3 event and was sustained for a further 20000 years.

It is my opinion that the Ursus spelaeus bones of Layer 3 were re-deposited at this she-

Itered site. They could therefore be significantly older than the 146000 +- 14000 "best date" from YUG K2R Base.

An alternative possibility is that the bones of Layer 3 are intrusions from Layer 5. If this is so, then the *Ursus spelaeus* could be younger than the Layer 4 "best date" of 126000 +- 11000 years. This question should be resolved by careful sedimentological studies at the site.

I belive that this is the first time that the ²³⁰Th: ²³⁴U dating method has been applied to a problem in the stratigraphy of *Ursus spelaeus*. The Križna Jama site is clearly of great interest and may yield further results in the future.

U series dating of speleothems by mass spectrometry

In 1988–89 our laboratory obtained the first ²³⁰Th: ²³⁴ U dates on speleothems by means of direct atom counting in a mass spectrometer (Li et alia 1989). This new method requires access to an extremely expensive instrument (a solid source mass spectrometer). However (i) it greatly increases the precision of the date (the first standard deviation is normally less than one per cent of the age determined), (ii) it extends the potential dating range back to 500000 or even 600000 years BP, and (iii) it requires much smaller samples for analysis. All of these features should permit us to obtain better results upon the Križna Jama specimens that still remain in the McMaster lab. I hope to undertake the work during 1990, and will report it to you; I deeply regret that Rado cannot take part in this exciting new development.

Acknowledgements

Mrs. Maja Kranjc assisted Dr. Gospodarič at the collection site. The cost of U series analyses at McMaster University was supported by a research grant to Ford from the Natural Sciences and Engineering Research Council of Canada.

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Z U SERIJO DOLOČENE STAROSTI PLASTI Z JAMSKIM MEDVEDOM IZ KRIŽNE JAME, SLOVENIJA

Povzetek

Zaradi nizke vsebnosti urana in visoke detritične in organske kontaminacije in možnega razpadanja urana so sedimenti jamskega medveda iz Križne jame zelo zapleteni za običajno analizo U z alfa spekrometrijo. Takšni sedimenti so običajno na arheoloških najdiščih ob jamskih vhodih ali na lehnjaku. Za tovrstne sedimente je Schwarcz (1980) predlagal izohrono datacijsko tehniko, če ponovljene analize zelo umazanih vzorcev dajejo dobro linearno povezanost razmerij, ²³⁰Th: ²³²Th v ²³²U:²³⁴Th je naklon indikativen za pravo starost vzorca. Na sliki 2 je predstavljena izohrona slika za vse vzorce iz tabele 1. Dobro linearno razmerje je opazno. Naklon linije daje izohrono srednjo starost 150000 let BP za celotno zbirko.

Naše mnenje je, da stratigrafsko serijo podatkov, ki smo jih dobili iz kapnikov "April 1982" sprejmemo, kot je predstavljena na tabeli 1. Vzorec YUG4K predstavlja sigo v plast 2., vzorec JUG K2R pa sigo v plasti 4. Ali so bili vzorci narobe označeni, ali pa se je zgodila napaka pri vzorčevanju v jami.

Zaporedje podatkov in sedimentologija vzorčnega mesta nam dasta naslednje podatke:

- 1. Rast sige v plasti 2 se je končala okrog 146.000 BP s poplavo ali je bila prekrita s sedimenti, ki so vsebovali kosti in kostne delce jamskega medveda.
- 2. Trajanje odlaganja meljaste plasti 3 je bilo prekratko, da bi ga lahko določili z U datacijo. Mogoče je bila le ena, zelo velika poplava. Kratkost tega dogodka dodatno prispeva k težavam, kako ločiti sigo, ki se je odlagala pred in po njem.
- 3. Odlaganje sige v plasti 4 se je začelo na vrhu tega melja pred približno 146000 in se je nadaljevalo do 126000 BP. Končalo se je s hitrim odlaganjem melja s kostmi jamskega medveda = meljasta plast 5.
- 4. Odlaganje kalcita se je nadaljevalo v plasti 6, vendar je vanj stalno dotekal melj iz zraka ali vode in kontaminiral vzorec.
- 5. Vrh plasti (plast 6) je odrezan zaradi poplave. Mešani klastični sedimenti plasti 7 so se odlagali na prisekano površino po tem dogodku. Vzorci "April 1982" U serije predvidevajo približno starost 146000 let za plast 3, kar je podprto tudi z rezultati izohronega testa iz U serij JUG K1A Ponovitev, YZG K2A Ponovitev ("popravljena" starost) in KYU 4S dno. YUG K4 v sredini, KYU 2 dno in KYU 2H vzorci so dali sprejemljive rezultate, ki potrjujejo, da je siga plasti 2 rasla, z enim ali dvema presledkoma, od 20.000 do 40.000 let pred odložitvijo plasti 3. Podatki o YUG K2R sredina in vrh nam pokažejo enak način odlaganja kalcita kot pri plasti 3.

Moje mnenje je, da so bile kosti jamskega medveda v plasti 3 ponovno odložene na tem zaščitenem mestu. Zato so lahko precej starejše od 146.000 *-14.000 "najboljši podatek" iz YUG K2R dna.

Druga možnost je, da so prišle kosti v plast 3 iz plasti 5. Če je tako, potem bi bil jamski medved lahko mlajši od "najboljšega podatka" v plasti 4, ki je 126.000 +- 11.000 let. To vprašanje lahko razreši skrbna sedimentološka študija na licu mesta.

Mislim, da je bila v tem primeru prvič uporabljena ²³⁰Th:²³⁴U metoda za reševanje problema stratigrafije jamskega medveda. Križna jama je zelo pomembno najdišče in v bodočnosti lahko računamo z dobrimi rezultati.