
Research Communication

Dating Archaeological Bone Specimens Using Natural Gamma Emitter Radionuclides

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Summary: Gamma ray emitter elements in local cave-soils and buried bone are assessed to attempt to establish a rôle for the use of gamma ray spectrometry for dating of buried bones found in an archaeological context. Thorium[234] > Potassium[40] ratio measurements are suggested to be possible non-destructive means of dating archaeological skeletal remains.

Keywords: radionuclides, gamma-emitters, archaeology, skeletal

Introduction

The first, and in some ways the most important, step in much archaeological research involves ordering things into chronological sequences. These sequences can then be used for relative dating. Careful attention to stratigraphy during archaeological excavations is essential for establishing the chronological sequence of the deposits. Problems with stratigraphical chronology may however occur in cases where there may have been physical movement of deposits or in burial deposits. A useful method of assessing whether several bones found in association are in fact of the same relative age is chemical and radiometric dating by studying nitrogen, fluorine and uranium content (Renfrew and Bahn, 1994).

The concentration of nitrogen in buried bone progressively decreases with time, while the concentration of fluorine and uranium increase with antiquity (Bowen, 1958). The loss of nitrogen in buried bone is dependent on the temperature and the water, chemical and bacteriological content of the environment in which the bone is buried. Similarly, the uptake of uranium and fluorine by buried organic remains is related to chemical factors in the soil deposit (Leute, 1987). The described methods to assay these elements in buried bones require either the partial destruction of the specimen or the use of an alpha-particle spectrometer. The present study attempts to elucidate the Maltese phreatic cave system dynamics pertaining to Uranium[238] daughter elements, and to correlate C14-dated bone with the gamma emitter daughter radionuclides of uranium using a simple gamma-ray spectrometer without the need of destroying the specimens.

Material and Methods

²³⁴Thorium and ²²⁶Radium, daughter elements of ²³⁸Uranium present in soil, are gamma ray emitters. ⁴⁰Potassium is another gamma ray emitter present in soil and rock (IAEA, 1989). A high resolution gamma ray spectrometry system can be used to identify gamma-emitting radionuclides with energies ranging from 60 keV to 2 MeV, depending on the type of detector, in a large variety of sample matrices (IAEA, 1989). The

simultaneous detection of several gamma radionuclide emitters in the sample material was carried out with a high resolution detector (coaxial germanium diode) connected to a multichannel analyser. Automatic processing of the collected spectral data was controlled by a computer system.

To assess the chemical dynamics of a phreatic limestone cave (Ghar Hasan) in Malta, gamma ray spectrometry was performed on cave rock, percolating water, recent cave-floor soil, and Pleistocene roof-soil. The results of these specimens were compared with soil obtained from the cliffs above the cave. Mammalian bone, also found buried in the cave, was similarly assayed. This bone was considered to be of very recent origin (probably less than a decade) in view of the good anatomical preservation of the individual bone components and the consistency of the soil in which the animal had been buried. The assays of the cave-floor earth samples obtained from Ghar Hasan were compared to further specimens obtained from other phreatic caves in Malta (Ghar il-Friefet and Ghar Dalam both at Birzebbugia).

Bones of varying antiquity obtained from various sites were further analysed for gamma-emitting radionuclides. The archaeological human bone specimens obtained from Fleur des Lys and BurMghez burials were radiocarbon dated at Oxford University and the British Museum by one of the authors (AM). The Fleur des Lys specimen [BM-3015] gave a date of 2500 years BP [Mifsud and Mifsud, 1997]. The BurMghez specimen [OxA-8165], presently conserved at the British Museum, yielded a date of 5300 years BP (Mifsud 1999). Pleistocene undated cervine fossil remains from Ghar Dalam from a private collection [AM/Vb:1-2] originating from the Carnivora Stage layer of the floor deposit and a hippopotamus fragment [AM/VII:1] originating from the Gliridae Stage layer were also similarly assayed (Zammit-Maempel, 1989; Savona-Ventura and Mifsud, 1998).

For the purposes of the present investigation, this first daughter radionuclide ²³⁴Th was considered to be more closely representing the levels of ²³⁸U than the other

daughter radionuclides. This assumption was based on the very short half-life of this radionuclide (24 days in contrast to the half-life of ^{226}Ra 1600 years). Since absolute values vary according to the type of specimen, the results obtained for the various samples were standardised as a ratio to ^{40}K . It was assumed that the high solubility of potassium salts results in equilibrium values of that radionuclide in all samples.

Results

The Ghar Hasan cave system yielded a series of $^{234}\text{Th}/^{40}\text{K}$ and $^{234}\text{Th}/^{226}\text{Ra}$ ratio results (Table 1). The $^{234}\text{Th}/^{40}\text{K}$ ratio of the percolating water (0.94) was higher than the ratio obtained for the cave-floor soil (~0.45) and even higher than the ratio obtained for the cave-roof soil (0.39) and the superficial cliff soil (0.26). Radium salts in limestone deposits appear to be markedly more soluble than Thorium salts, so that the soil samples had higher $^{234}\text{Th}/^{226}\text{Ra}$ ratios (1.22-1.79) than the cave rock (0.42). The recently buried bone samples appear to have equilibrated with the percolating water showing approximately similar values of $^{234}\text{Th}/^{226}\text{Ra}$ ratios (0.79 and 0.73 respectively). There appears to be an active absorption of Thorium by bone resulting in an elevation of $^{234}\text{Th}/^{40}\text{K}$ ratio (1.61 vs 0.94 in water).

Ghar Hasan SPECIMENS	$^{40}\text{Potassium}$ Bq/g	$^{234}\text{Thorium}$ Bq/g	$^{226}\text{Radium}$ Bq/g	$^{234}\text{Thorium}$ / $^{40}\text{Potassium}$	$^{234}\text{Thorium}$ / $^{226}\text{Radium}$
Superficial Cliff soil	5.90E-01	1.54E-01	1.26E-01	0.26	1.22
Cave Rock	1.57E+01	9.99E+00	2.39E+01	0.63	0.42
Percolating Water	3.35E+00	3.20E+00	4.43E+00	0.94	0.73
Recent Cave-floor soil (2 sites)	2.57E-01 3.03E-01	1.13E-01 1.36E-01	6.97E-02 7.60E-02	0.44 0.45	1.61 1.79
Pleistocene Cave-roof soil	9.69E+01	3.78E+01	3.03E+01	0.39	1.25
Recent buried mammalian bones	1.22E-01	1.96E-01	2.47E-01	1.61	0.79

Table 1: Ghar Hasan system

The cave-floor soil samples (Table 2) obtained from the three sites in Malta show that similar factors are operative in various phreatic cave systems, with the $^{234}\text{Th}/^{40}\text{K}$ ratios being approximately equal in soil samples from the three caves; this gave a mean of $0.422 + 0.052\text{sd}$ (range 0.32 - 0.47). The mean $^{234}\text{Th}/^{226}\text{Ra}$ ratio showed a wider variation in result with a range of 0.72 - 1.79 (mean = $1.19 + 0.396\text{sd}$).

The ratios obtained for the various buried bony remains suggest that bones initially equilibrate with the

Cave soil SPECIMENS	$^{40}\text{Potassium}$ Bq/g	$^{234}\text{Thorium}$ Bq/g	$^{226}\text{Radium}$ Bq/g	$^{234}\text{Thorium}$ / $^{40}\text{Potassium}$	$^{234}\text{Thorium}$ / $^{226}\text{Radium}$
Ghar Hasan Cave-floor soil (2 sites)	2.57E-01 3.03E-01	1.13E-01 1.36E-01	6.97E-02 7.60E-02	0.44 0.45	1.61 1.79
Ghar Hasan Pleistocene Cave-roof soil	9.69E+01	3.78E+01	3.03E+01	0.39	1.25
Ghar il-Friefet Cave-floor (2 sites)	2.84E-01 4.29E-01	9.15E-02 2.01E-01	1.28E-01 2.36E-01	0.32 0.47	0.72 0.85
Ghar Dalam Pleistocene Cave-floor soil	4.46E-01	2.04E-01	2.18E-01	0.46	0.94
Mean + sd				0.422 + 0.052	1.190 + 0.396

Table 2: Cave-floor soils

Buried Bone SPECIMENS	$^{40}\text{Potassium}$ Bq/g	$^{234}\text{Thorium}$ Bq/g	$^{226}\text{Radium}$ Bq/g	$^{234}\text{Thorium}$ / $^{40}\text{Potassium}$	$^{234}\text{Thorium}$ / $^{226}\text{Radium}$
Percolating Water				0.94	0.73
Cave Soil Mean + s.d.				0.422 + 0.052	1.190 + 0.396
Mgar Cave (surface find: recent)	5.93E-02	0	6.02E-02	0	0
Ghar Hasan (buried: recent)	1.22E-01	1.96E-01	2.47E-01	1.61	0.79
Fleur de Lys burial (C^{14} : 2500 yrs BP)	1.26E-01	5.81E-01	3.64E-01	4.61	1.6
BurMghez (C^{14} : 5300 yrs BP)	1.21E-01 1.29E-01	8.39E-01 1.11E+00	5.59E-01 5.05E-01	6.93 8.61	1.5 2.19
Mean + sd				7.77 + 1.19	1.85 + 0.49
Ghar Dalam Cervus fossil LAYER Vb	2.60E-01 1.80E-01	1.96E+00 1.42E+00	1.12E+00 7.59E-01	7.54 7.89	1.75 1.87
Mean + sd				7.72 + 0.18	1.81 + 0.06
Ghar Dalam Hippopotamus fossil LAYER VII	2.12E-01	2.07E+00	1.04E+00	9.76	1.99

Table 3: Bone samples

percolating water, so that the $^{234}\text{Th}/^{226}\text{Ra}$ ratio of the bone approximates that of percolating water. With increasing antiquity including that of Pleistocene fossils, the bone $^{234}\text{Th}/^{226}\text{Ra}$ ratio appears to approximate the value of the cave-floor soil. Recently buried bone appears to absorb Thorium actively, thus increasing the $^{234}\text{Th}/^{40}\text{K}$ ratio, from 0.94 in percolating water to a 1.61 value in recently buried bone (Table 1). With increasing antiquity, the $^{234}\text{Th}/^{40}\text{K}$ ratio of buried bone appears to increase progressively to give a $^{234}\text{Th}/^{40}\text{K}$ ratio of 4.61 after 2500 years, and a mean of $7.77 + 1.19\text{sd}$ after 5300 years (Table 3). The $^{234}\text{Th}/^{40}\text{K}$ ratio results obtained for the Pleistocene remains appeared similar to those of the Neolithic remains, with the younger cervine bones having a mean value of $7.72 + 0.16\text{sd}$, and the older hippopotamus bone a value of 9.76.

Discussion

Buried bone equilibrates with its environment and undergoes active chemical changes, which reflect its antiquity. Thus with increasing age, the nitrogen content of buried bone progressively decreases at a rate dependent on the temperature and the water, and the chemical and bacteriological content of the environment in which the bone is buried (Protsch, 1986). At the same time, percolating ground water has significant effects on the composition of bone. Elements present in solution in the ground water - fluorine, uranium and iron - are absorbed gradually by the bone, their levels increasing with increasing antiquity. The rate of increase in fluorine, uranium and iron depend on the local concentrations of the elements in the percolating water and the rate of water flow (Protsch, 1986). Modern bone has only traces of these elements, with the level of uranium oxide being practically nil, fluorine being less than 0.1%, while iron amounts to about 0.007% (Leute, 1987; Diem and Lentner, 1975). The uptake of uranium by buried organic remains is related to chemical factors in the soil deposit, the rate of water flow and the concentration of the elements in the percolating water. Because local chemical factors can vary, the chemical tests cannot be used for the basis of

an absolute dating test. However on an individual site or in sites shown to have similar chemical characteristics, chemical dating can distinguish bones of different age found in apparent stratigraphical association (Protsch, 1986).

The three major naturally occurring primordial radionuclides usually present in soil and rock include the isotopes of uranium [^{238}U] and thorium [^{232}Th] plus their daughters, and an isotope of Potassium [^{40}K]. Native uranium contains three isotopes: 99.28% ^{238}U , 0.7% ^{235}U , and 0.006% ^{234}U . Natural isotopes of uranium are alpha emitters with very long half-life (4.5×10^9 years for ^{238}U). The ^{238}U series produces a number of gamma emitter radionuclides, notably ^{234}Th , ^{226}Ra , ^{214}Pb and ^{214}Bi . The Radium - Uranium ratio in natural compounds is 3.4×10^{-7} . ^{234}Th and ^{226}Ra are beta and alpha emitters respectively, with gamma ray emission as a by-product of the surplus excitation energy. The amount of radionuclides varies according to the type of rock or soil formation found in the locality, being generally higher in volcanic regions. In a region where limestone predominates, such as the Maltese Islands, the radionuclide levels are low but of sufficient levels to allow the use of radionuclide levels to be used for archaeological relative and absolute dating (IAEA, 1989; Songina, 1970).

Buried bones and teeth are exposed to the action of the percolating ground water containing uranium salts in solution. The uranium irreversibly substitutes the phosphate content of bone, mainly the hydroxy-apatite. Fossil bones had been shown to contain uranium by Lord Rayleigh in 1908, but the technique was only revived and adapted in 1955 by Davidson and Bowie at the Atomic Energy Division of the Geological Society. It was established that although the uranium oxide concentration varied in different localities, the amount increased proportionately with its antiquity. Specimens in the same environment varied directly in proportion with the length of time that they were buried (Davidson and Bowie, 1955). Levels of uranium oxide in modern bone is practically nil, but in ancient buried bone these may rise to levels as high as 1000 ppm, depending on the concentration of uranium oxide in the percolating water. The range in fossil bone has been reported as lying between 1 and 1000 ppm (Leute, 1987; Aitken, 1990), the level increasing proportionately with its antiquity (Davidson and Bowie, 1955).

The level of uranium oxide and its daughter elements have thus been noted to vary in different localities depending on the surrounding rock strata and leaching effects (Bowen, 1958; Oakley, 1970). In spite of these expected differences in different geological formations, it is expected that the uranium oxide and daughter element concentrations should be similar in matching closed system environments. Karstic limestone cave environments are such situations, where the formative and erosive processes are approximately similar in their low levels of uranium salts (Bowen, 1958; Oakley, 1970). It would be expected that the concentration of uranium oxide and daughter elements in cave floor soil would equilibrate in a similar fashion, provided that the

cave floor soil is of a similar age.

The different cave soils studied showed a relatively stable $^{234}\text{Th}/^{40}\text{K}$ ratio, with a mean value of $0.422 \pm 0.052\text{sd}$. Potassium is an unrelated gamma emitter which, because of the high solubility of potassium salts, may be assumed to be in a steady state equilibrium in all samples. The stable $^{234}\text{Th}/^{40}\text{K}$ ratio in Maltese karstic caves, including Ghar Hasan, Ghar Dalam and Ghar il-Friefet, reflects the steady state relationship between these two gamma emitter elements in cave floor soil. The similarity of the results obtained from the different sites suggests that bony remains buried in karstic limestone cave systems in Malta may be compared since they are exposed to near identical chemical and physical conditions. The $^{234}\text{Th}/^{226}\text{Ra}$ ratios in the different cave soils appeared to show a wider variation in results around a mean value of $1.19 \pm 0.396\text{sd}$, with a range of 0.72-1.79. The reasons for this wide difference range in the daughter elements of uranium have not been elucidated, but may reflect the possible age of the soil sample with older soils showing a lower ratio. The difference in the half-lives of the two radionuclides - ^{234}Th : 24 days; ^{226}Ra 1600 years - would result in higher Radium levels and thus a lower $^{234}\text{Th}/^{226}\text{Ra}$ ratio in older specimens. Further studies in this regard are required.

A dynamic interplay of factors would be expected to exist in any cave system, such as the one exemplified by Ghar Hasan. The cave-soil samples originated from the cave rock, and should have had an original value equivalent to the $^{234}\text{Th}/^{40}\text{K}$ ratio of the rock (0.63). The ratios in the various cave-soil samples have been apparently modified over time through the action of percolating rainwater. Rainwater is acidic in nature and has a near zero $^{234}\text{Th}/^{40}\text{K}$ ratio. This falls onto the superficial soil/rock dissolving and leaching away the various elements at variable rates depending on the solubility. With prolonged exposure, the acid soluble elements are leached away, leaving the terrarossa soil typical of the superficial cliff in which Ghar Hasan is found (Schembri and Baldacchino, 1992). As a result of this prolonged leaching, the $^{234}\text{Th}/^{40}\text{K}$ ratio of the superficial soil (0.26) was very much lower than the $^{234}\text{Th}/^{40}\text{K}$ ratio of the derivative cliff rock (0.63). The minerals are thus continuously leached into the percolating water, which showed a higher $^{234}\text{Th}/^{40}\text{K}$ ratio (0.94). This water with a relatively high $^{234}\text{Th}/^{40}\text{K}$ ratio leaches the cave-floor and cave-roof soils, which also originated from the same derivative rock ($^{234}\text{Th}/^{40}\text{K}$ ratio 0.63), and reduces the $^{234}\text{Th}/^{40}\text{K}$ ratio of these soils to a lesser extent (0.44-0.46) than the superficial cliff soil. The presumably recent bony remains buried in the cave-floor soil equilibrate with the percolating water and concentrate uranium and its daughter elements by an active process, whereby the phosphate content of hydroxy-apatite in bone is replaced. This results in a high $^{234}\text{Th}/^{40}\text{K}$ ratio (1.61) [Figure 1].

The $^{234}\text{Th}/^{40}\text{K}$ of the bones appear to progressively increase with increasing antiquity, thus reflecting the active binding process of the bone with uranium series elements. The BurMghez specimens, deposited in the British Museum after their excavation in the early

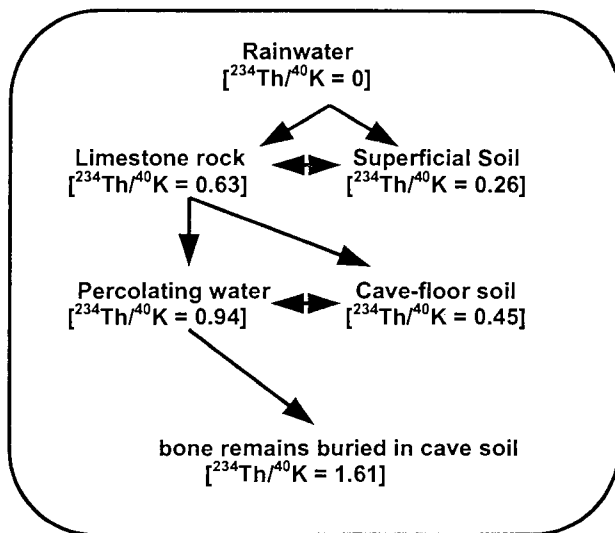


Figure 1: Ghar Hasan Cave system dynamics

twentieth century, had been dated, on the basis of pottery shards correlation, to the Ggantija Phase, circa 3600-3000 BC, or circa 5600-5000 BP (Evans, 1972). Carbon dating carried out at Oxford has confirmed a date of 5300 years BP for these bony remains. The $^{234}\text{Th}/^{40}\text{K}$ ratio for the BurMghez bones (mean $7.77 \pm 1.19\text{sd}$) correlated very well with the established artifactual and carbon-14 dating, thus giving the highest reading. The Fleur des Lys specimens had been previously assayed for nitrogen percentage by the Natural History Museum, using the Weiler and Strauss unwashed technique. The nitrogen percentage for repeated tests varied from 0.43-2.58% [Bone Analysis Malta Samples Ma.27]. This very wide range for the same specimens reflects the unsuitability of relying solely on nitrogen assay as an index of antiquity (Mifsud and Mifsud, 1997). Carbon dating of these remains [BM-3015] gave a date of 2500 years BP. This date correlated with the $^{234}\text{Th}/^{40}\text{K}$ ratio obtained for these specimens (4.61), which gave a value approximately midway between the BurMghez and recent Ghar Hasan remains. The results suggests that the increase of $^{234}\text{Th}/^{40}\text{K}$ ratio in archaeological bones appears to be directly proportional to age, increasing at a rate approximating 1.5 per 1000 years of burial [Figure 2]. The process appears to eventually become saturated so that the $^{234}\text{Th}/^{40}\text{K}$ ratios of Pleistocene fossils remained approximately the same, or increased minimally, from the value obtained for the Neolithic remains. Definite dating of the Pleistocene layers at Ghar Dalam has yet to be undertaken. The only date is that obtained for a hippopotamus fossil dated by electron spin resonance and uranium series disequilibria to 130,000-110,000 years BP (Bouchez et al, 1988). Uranium oxide levels of cervine and hippopotamus bones have been previously carried out by the Natural History Museum, London. These gave values of $7.67 \pm 4.04\text{sd}$ for cervine bones and $6.50 \pm 3.54\text{sd}$ for hippopotamus bones (Mifsud and Mifsud, 1997; Savona-Ventura and Mifsud, 1998). The exact stratigraphical context of these tested fossils had not been defined.

The $^{234}\text{Th}/^{226}\text{Ra}$ ratios are more difficult to interpret, but appear to reflect the age of the specimens. ^{234}Th has a

very short half-life (24 days) contrasting with the half-life of ^{226}Ra (1600 years). This would result in a gradual increase in the radium concentration with time. With increasing age however, the radium would also significantly break down to other radionuclides, further altering the $^{234}\text{Th}/^{226}\text{Ra}$ ratios of very old deposits. The $^{234}\text{Th}/^{226}\text{Ra}$ ratio for water approximated 0.73, while the cave-floor soil samples approximated 1.7. The cave-roof soil sample previously described as Pleistocene (Shaw, 1950; Shaw, 1953) gave a median $^{234}\text{Th}/^{226}\text{Ra}$ figure of 0.94. The recent bone samples appeared to be in equilibrium with the percolating water, while with increasing antiquity the $^{234}\text{Th}/^{226}\text{Ra}$ ratio of buried bone (1.5-2.19) approximates the ratio of cave-floor soil.

The present study suggests that the $^{234}\text{Th}/^{40}\text{K}$ ratios of various bone specimens collected from various caves in Malta show a definite trend in values, the ratio increasing with increasing antiquity of the bone sample. The measurement of $^{234}\text{Th}/^{40}\text{K}$ ratio may thus be useful in correlating the bone specimen to stratigraphy, and can be used for relative dating in an archaeological context. It does not however appear to be a useful dating method for fossil specimens. The present study can be considered only as a pilot study in view of the small number of carbon-dated bone specimens. Further analysis of carbon-14 dated bones is necessary to confirm the suitability and reliability of this technique. Further sources of error in the technique need to be investigated. Errors may be caused by improper spectral identities, changes in background, errors in calibration of various geometries, and lack of homogeneity in samples (IAEA, 1989). Since the samples tested in this study were not in a standardised geometry, some variation in the readings could be expected due to the difference in sample homogeneity. This technical drawback was partly overcome, where possible, by using samples of approximately equal weight and size. If confirmed as useful, the use of gamma ray spectrometry for dating of archaeological skeletal remains has the advantage of being a non-destructive technique which preserves the integrity of the specimen.

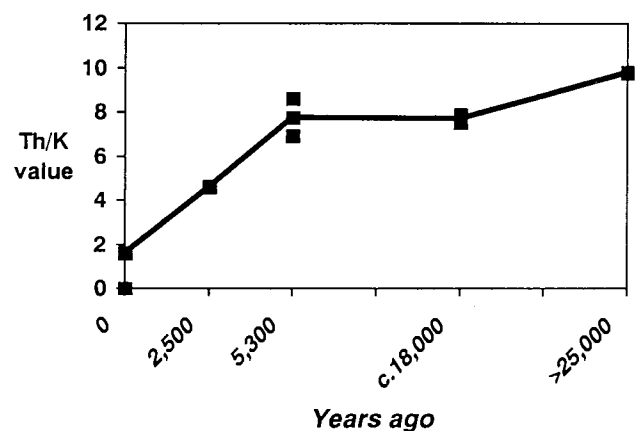


Figure 2: Bone Th/K ratio with increasing antiquity

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