APPENDIX 2 ISOTOPIC ANALYSIS OF THE HUMAN TEETH

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A.2.1 Background

Samples of tooth enamel, retrieved from the Parliament House and St Giles' excavated assemblages, were submitted to the NERC Isotope Geosciences Laboratory (NIGL) for strontium ($^{87}Sr/^{86}Sr$) and oxygen ($\delta^{18}O$) isotope analysis for population migration data. Details of methodologies are fully recorded in the site archive report at the National Record of the Historic Environment of Scotland, where a full report on the findings can also be found. In this report PH SK refers to Parliament House Skeleton, while PH-81 refers to St Giles' Skeleton.

Fourteen tooth enamel samples were submitted for analysis. The samples were in a range of preservation states. The majority of the teeth were in a satisfactory condition for analysis and, following cleaning, most of the enamel samples were hard and translucent, a sign of good preservation. Several samples had poor preservation with heavily cracked and friable enamel. As a result, one enamel sample was not analysed (PH SK 44).

A.2.1.1 Local bedrock geology

This area of Lothian is dominated by rocks of the Carboniferous period and the bedrock is Carboniferous Limestone with a large area of andesitic/basaltic volcanic rocks nearby. To the south, the Southern Uplands are dominated by Palaeozoic meta-sedimentary rocks, and to the north, beyond the Devonian deposits, the older Scottish Proterozoic rocks crop out.

A.2.1.2 Background to oxygen and strontium isotopic analysis of human remains

Oxygen and strontium isotopes are fixed in enamel biogenic phosphate at the time of tooth formation. Biogenic phosphate is extremely robust and the isotopic signature of enamel does not change during life, nor is it altered in the burial environment. Oxygen isotopes are derived primarily from ingested fluids and reflect the isotopic value of available meteoric/ground/drinking water. Strontium isotopes are derived from both solid and liquid food and relate to the soil-derived bio-available strontium which, in the absence of any surficial deposits such as peat, loess, tills etc, is related to the geology of the area where the food was produced. As strontium and oxygen isotopes behave independently of one another, they allow two parameters for investigating an individual's place of origin and migration patterns.

A.2.2 Analytical methods

A.2.2.1 Sr isotopes

The samples were cleaned ultrasonically for five minutes in high purity water and rinsed twice to remove loosely adhered material. The available enamel surface of the teeth was abraded from the surface to a depth of >100 microns using a tungsten carbide dental bur and the removed material discarded. Thin enamel slices were then cut from the tooth using a flexible diamond edged rotary dental saw. All surfaces were mechanically cleaned with a tungsten carbide bur to remove adhering dentine. The resulting samples were transferred to a clean (class 100, laminar flow cabinet) working area for further preparation. In the clean environment, the samples were first cleaned ultrasonically in high purity water to remove dust, rinsed twice, dried down in high purity acetone and then weighed into pre-cleaned Teflon beakers. The samples were mixed with ^{84}Sr tracer solution and dissolved in Teflon-distilled 16M nitric acid (HNO_3). Strontium was isolated using conventional Dowex resin. Following elution from the column, each strontium sample was loaded onto a single Re Filament with TaF, following the method of Birck (1986), and the isotope composition and concentrations were determined by Thermal Ionisation Mass Spectroscopy (TIMS) using a Finnigan Triton multi-collector mass spectrometer. The international standard for $^{87}Sr/^{86}Sr$, NBS987, gave a value of 0.710284 ± 0.000010 (n=20, 2SD) for static analysis. All strontium ratios have been corrected to a value for the standard of 0.710250. Blank values were in the region of 100pg. Data are presented in Table 8.

Table 8 Strontium isotope and concentration data and oxygen isotope data for tooth enamel and environmental samples from Parliament House and St Giles' Cathedral (This table presents the strontium concentration, $^{87}Sr/^{86}Sr$ isotope composition, and the phosphate oxygen isotope composition in the form of $\delta^{18}O_{VSMOW}$ (‰), which is converted to a drinking water value (Mean $\delta^{18}O_{dw}$ (‰). * Preservation rating based on Montgomery (2002).)

Sample	Sr ppm	$^{87}Sr/^{86}Sr_n$	Mean $\delta^{18}O_{VSMOW}$ (‰)	Mean $\delta^{18}O_{dw}$ (‰)	Preservation (1 = good, 6 = poor)*	Comment
St Giles' period 3						
PH-81 SK 4	109	0.71143	17.5	-7.3	4.5	root present
PH-81 SK 10	173	0.70936	18.4	-5.3	3.5	root present
PH-81 SK 11	347	0.70976	17.1	-8.0	4.0	root present
PH-81 SK 16	133	0.70958	18.2	-5.7	3.0	root present
PH-81 SK 18	168	0.70945	19.8	-2.1	4.0	root present
PH-81 SK 21	146	0.70962	18.4	-5.3	3.0	root present
PH-81 SK 48	223	0.70961	17.7	-6.8	4.0	root present
Parliament House						
PH SK 16	341	0.70916	17.0	-8.4	4.0	single root, worn, juvenile?
PH SK 45	219	0.70969	17.1	-8.0	4.0	unerupted
PH SK 58	91	0.71006	18.8	-4.3	4.0	root, worn discoloured
PH SK 60	87	0.70983	17.3	-7.7	3.5	root, worn discoloured
PH SK 71	77	0.71646	17.4	-7.3	3.0	root
PH SK 92	92	0.70905	17.7	-6.8	3.5	root, worn discoloured
Environmental samples						
PH SK 25s		0.70929				
PH SK 60d	237	0.70948				
PH SK 16d	280	0.70921				
PH-81 SK 11d	229	0.70947				
PH-81 SK 16d	168	0.70931				

A.2.2.2 Oxygen isotopes

Silver phosphate was precipitated using the method of O'Neil et al (1994), from small fragments of clean enamel (15–20mg). The fragments of enamel were cleaned in concentrated hydrogen peroxide (H_2O_2) for 24 hours to remove organic material and subsequently the H_2O_2 was evaporated to dryness. The samples were then dissolved in 2M nitric acid and transferred to clean polypropylene test tubes. Each sample was then treated with 2M potassium hydroxide and 2M hydrogen fluoride to remove calcium from the solution by precipitation. The samples were then centrifuged and the supernatant added to beakers containing ammoniacal silver nitrate solution and heated gently to precipitate silver phosphate. The silver phosphate was filtered, rinsed, dried and weighed into silver capsules for analysis. Oxygen isotope measurements on each sample were carried out in triplicate by thermal conversion continuous flow isotope ratio mass spectrometry (TC/EA-CFIRMS). The reference material, NBS120C, calibrated against certified reference material NBS127 (assuming $\delta^{18}O$ of NBS127 = +20.3‰ versus SMOW; IAEA 2004), has an accepted value of 21.70‰ (Chenery 2005). The reproducibility of NBS120C during this set of analyses was 21.64‰ ± 0.26 (1σ, n=54). Drinking water values are calculated using Levinson's equation (Levinson et al 1987), after correction for the difference between the average published values for NBS120C used at NIGL and the value for NBS120B used by Levinson. Data are presented in Table 8.

A.2.3 Results

A.2.3.1 Strontium isotopes

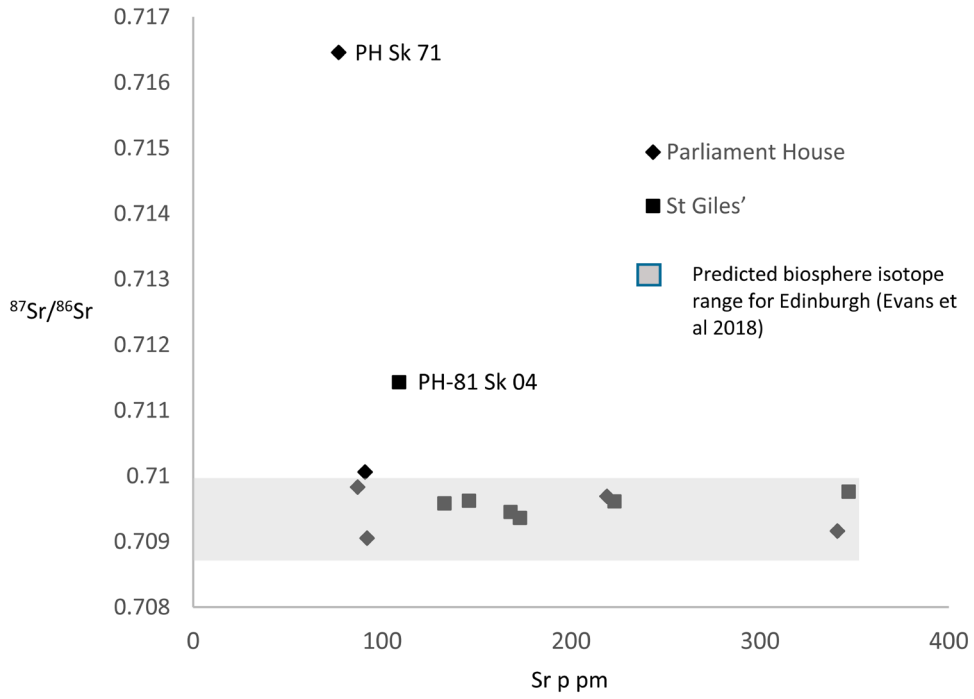
The site of Parliament House in Edinburgh is close to the coast and is underlain by limestones, sandstones and Coal Measure sequences of the Carboniferous Period. The soil leach and dentine (re-equilibrated with pore fluids during burial) give a data range 0.7092–0.7095 as recorded by the leach of soils in environmental samples provided from the burial site (Table 8), and from the dentine, which will equilibrate after burial. These values are within the predicted interquartile range of $^{87}Sr/^{86}Sr$ = 0.7088–0.7101 for biosphere values in the area of Edinburgh (Evans et al 2018). The enamel from three burials from Parliament House form a group with *c* 100ppm Sr and $^{87}Sr/^{86}Sr$ ratios of 0.7091–0.7100 (PH SK 58, 60 and 92) (Illus 24). PH SK 71 has a similar strontium concentration but a much higher, more radiogenic $^{87}Sr/^{86}Sr$ signature of 0.71646. This very radiogenic value would exclude this individual from having spent their childhood in the Edinburgh area and the closest place that cannot be excluded as a possible site of childhood origin is the area of the Scottish Highlands near Ballater, which is *c* 110 miles north of Edinburgh. The samples from PH SK 16 and SK 45 are an un-erupted molar with only partially mineralised enamel, and a poorly preserved tooth from a juvenile. Neither is considered likely to preserve a life signal and the $^{87}Sr/^{86}Sr$ values from these two samples are considered to be re-equilibrated with the burial environment.

The St Giles' site yields one outlier, but otherwise these samples give a restricted range of $^{87}Sr/^{86}Sr$ consistent with an Edinburgh childhood, with the outlier (PH-81 SK 04) giving an $^{87}Sr/^{86}Sr$ ratio of 0.71143 (Illus 25). The closest area that could generate such values is in the Southern Uplands, about 20 miles south of Edinburgh.

A.2.3.2 Oxygen isotopes (Table 8)

The Parliament House individuals have a normally distributed oxygen isotope composition of $\delta^{18}O_{phosVSMOW}$ = 17.29 ± 0.57‰ (2SD, n=5) which is consistent with data from individuals raised in eastern Britain ($\delta^{18}O_{phosVSMOW}$ = 17.2‰ ± 1.3, 2SD, n=83, Evans et al 2012). There is one outlier at this site (PH SK 58), which gives a higher value of $\delta^{18}O_{phosVSMOW}$ = 18.83‰. The data from the St Giles' site gives $\delta^{18}O_{phosVSMOW}$ = 17.86 ± 1.04‰ (2SD, n= 6). The larger uncertainty on these data, when compared to Parliament House, suggests a more diverse drinking water supply but this may simply be an artefact of the small sample numbers. This site also includes an individual with an outlying high point of 19.83‰ (PH-81 SK 18).

Regarding the two outlier oxygen values, PH SK 58 is within the 2SD range of east coast British oxygen ($\delta^{18}O_{phosVSMOW}$ = 17.2‰ ± 1.3, 2SD, n=83, Evans et al 2012), but would be a value more typical



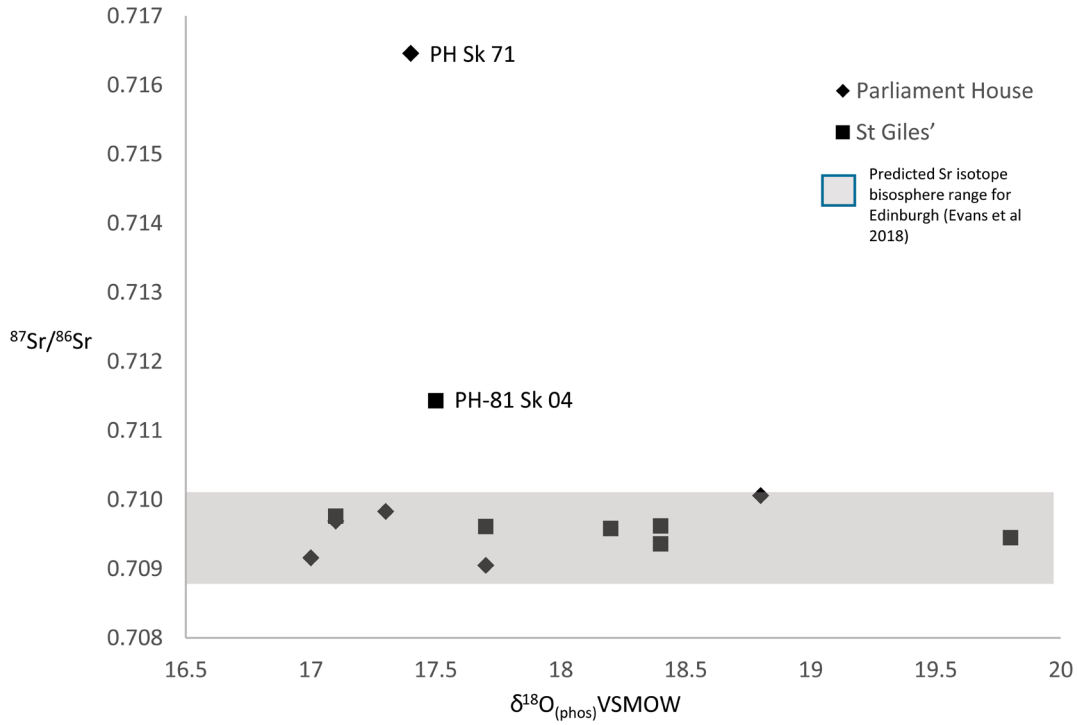
Illus 24 $^{87}Sr/^{86}Sr$ plotted against strontium concentration for samples from Parliament House and St Giles' (© AOC Archaeology Group)

of west coast sites. PH-81 SK 18, with its elevated value of $\delta^{18}O_{phosVSMOW}$ 19.83‰, is outside the 2SD range of values for west coast British data $\delta^{18}O_{phosVSMOW}$ = 18.2 ± 1.0‰ (2SD, n=40). The interpretation of these results, within the context of geographic origin, is based on the assumption that the main source of water into the body is from a drinking water source, derived from averaged rainwater compositions and typically of aquifer origin. If, for some reason, such a source is not the main water intake then the value can be modified. It is possible that either (i) the individual comes from a warmer climate where the Sr isotope systematics are indistinguishable from those in Lothian, or (ii), in the case of the single outliers, that the individual is a random statistical outlier in the population or (iii) that the water intake of these individuals was modified in some way such that it did not conform to the model of un-modified UK water source (Brettell et al 2012).

A.2.4 Conclusions

The individuals from the two burial sites in Edinburgh can be summarised thus: Parliament House is a burial site for individuals whose Sr isotopes are consistent with the local environment and whose Sr concentrations are consistent with those of most other UK archaeological studies. Their oxygen isotope composition is normally distributed and in agreement with estimates for human tooth enamel in eastern Britain ($\delta^{18}O_{phosVSMOW}$ = 17.2 ± 1.4‰, 2SD, n=615, Evans et al 2012). The group includes one outlier (PH SK 71) with an elevated $^{87}Sr/^{86}Sr$ ratio (Illus 25), which suggests a childhood spent on Palaeozoic or older rocks such as those found in the highlands of Aberdeenshire and Perthshire.

The St Giles' group is different in several ways. The majority of this group has a restricted $^{87}Sr/^{86}Sr$ isotope composition which is consistent with the local area, but they tend to have elevated Sr concentrations, such as those recorded from Machair dwelling individuals of the Outer Hebrides. The oxygen isotope composition of this group is unusual, and if they are taken to be a local group, on the basis of their Sr isotopes, their oxygen is inconsistent with local drinking water values – they record $\delta^{18}O$ values that give drinking water values typical of warmer climates. It is beyond the scope of this study to explain fully the unusual oxygen isotope features seen in the St Giles' group but it is suggested that this is related to the manner in which they obtained or processed their drinking water.



Illus 25 The fields of Parliament House and St Giles' data in $^{87}Sr/^{86}Sr$ vs $\delta^{18}O_{SMOW}$ highlighting the two main outlying samples PH SK 71 and PH-81 SK 04 (© AOC Archaeology Group)