



Palace Green Library excavations 2013 (PGL13)

Isotopic studies

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with an appendix on *Carbon and nitrogen isotopes in animals from Durham City* by Andrew Millard¹, Jessica Blesch¹, Margaret Scollan¹, Jordan Rex¹, Katherine.Ulewicz¹, and Darren Gröcke²

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In 2013 two mass-burial pits were discovered at the Palace Green Library (see *Excavation Report*) and within these the remains of 29 individuals were recovered or observed (see *Human Bone Report*). Two hypotheses were advanced as to the origins of these individuals, either they were victims of a 16th or 17th century epidemic, such as plague, or they were the remains of prisoners from the 1650 Battle of Dunbar who had died while incarcerated in Durham Cathedral and Castle. Isotopic analysis of strontium, oxygen and lead was commissioned to elucidate their geographical origins.

1. Principles

People who spend their childhood in a particular geographical region have specific combination of stable isotopes preserved in the enamel of their teeth (Budd et al. 2004). Unlike minerals in bone, tooth enamel is not remodelled during the lifetime of the individual, and the childhood signal is preserved. Tooth enamel is also resistant to alteration by the burial environment (Budd et al. 2000). Measurements made on tooth enamel represent an average over the years that it took to form, and the mineralisation mechanisms (Simmons et al. 2013) mean that this averaging cannot be avoided.

The principles of strontium isotope analysis have been described by Bentley (2006) and may be summarised as follows. Strontium is an element that has no known function in human metabolism but it is absorbed as a trace element via calcium metabolism, and is found primarily in teeth and bones. There is natural variation in the isotopic composition of strontium due to the formation of the isotope ⁸⁷Sr by radioactive decay of ⁸⁷Rb. The ratio of ⁸⁷Sr to ⁸⁶Sr in rocks varies with the initial strontium and rubidium contents and the age of the rocks. Strontium is released by weathering into the soil and is then taken up without any change in isotope composition by plants and the animals that eat them. Thus local food-chains will reflect the local geology in their ⁸⁷Sr/⁸⁶Sr ratio, and this will be transmitted to humans. In the past without large-scale long-distance transport of foodstuffs, most foods would have been local. Plants contain more strontium than animals, so the ⁸⁷Sr/⁸⁶Sr ratio in humans reflects the origin of plant foods more than animal foods (Burton and Wright 1995). The oceans have a specific ⁸⁷Sr/⁸⁶Sr ratio of 0.71092, so sea-spray onto land near the coast and the consumption of marine foods can alter the expected strontium isotope ratio (Montgomery et al. 2007).

Variations in oxygen isotope ratios (expressed as $\delta^{18}\text{O}$, the difference from a standard) in humans are primarily a reflection of variations in the composition of water in the diet, the majority of which is drinking water (Daux et al. 2008). Drinking water is almost always sourced locally and other dietary water is in foodstuffs, which will also derive from local water sources. The oxygen isotope composition of groundwater and precipitation depends on climatic factors such as temperature and altitude, but the geographical patterns have changed very little over the last 10,000 years and have been extensively mapped (Bowen and Revenaugh 2003; Darling 2004; Darling et al. 2003; Darling and Talbot 2003). Thus it is possible to correlate measurements on archaeological remains with the modern maps. However, the oxygen isotope composition of human tissues is systematically altered from dietary water by metabolic processes, and a calibration equation has to be used to calculate drinking water values from measurements in the phosphate of tooth enamel (Daux et al. 2008), which introduces uncertainty in the reconstructed drinking water values (Pollard et al. 2011). Other processes can also alter the oxygen isotope ratio. Breast-feeding infants have elevated $\delta^{18}\text{O}$ values (Wright and Schwarcz 1998), so to simplify

interpretation it is best to sample the enamel of teeth that form after weaning, such as the second molar (M2) used in this study which forms at 3.5-7.0 years of age (AlQahtani et al. 2010; Moorrees et al. 1963). Humans can also elevate the $\delta^{18}\text{O}$ value of their drinking water by processes such as boiling and brewing (Brettell et al. 2012), and this must be allowed for in interpretation.

Lead is a naturally occurring component of all environments, but its levels have been raised significantly by pollution over the last century. Natural lead has an isotopic composition which varies with the local geology due to the formation of three of the four lead isotopes by the radioactive decay of uranium and thorium (Albarède et al. 2012). Before the advent of lead metallurgy, human exposure was very low and this is reflected in low concentrations of lead in tooth enamel with very varied isotope ratios representing natural environmental variability (Budd et al. 2004b; Millard et al. 2014; Montgomery et al. 2010). The use of lead in artefacts has exposed humans to much higher levels, and concentrations in tooth enamel have increased, but the lead isotope composition has become much less varied due to the small variation in the isotope composition of lead ore sources in a region. This reduction in variation is known as 'cultural focussing' of lead isotope ratios (Montgomery et al. 2010). Millard et al. (2014) have identified that lead concentrations in enamel over 870 ppb are unequivocal evidence for exposure to anthropogenic lead. In the post-medieval period lead exposure was greater in urban areas than rural areas (Millard et al. 2014)

2. Hypotheses

If these are Scottish prisoners, then they can be expected to have been drawn from all over Scotland (Reid 2004:24-24) and even from Ulster (Furgol 1990:318). They will show diverse isotopic signals, compatible with the known ranges of these isotope systems in Scotland. Some will be rural and others urban in origin and therefore some will have high lead exposure and others low lead exposure.

If these are victims of an epidemic, they will be mostly residents of Durham, but may include some visitors to the city. The majority will have originated from the local area, but some residents and visitors may have come from further afield, including a small minority from Scotland. The group is expected to show a limited range of isotopic ratios. Residents of the city are likely to be in the majority and they will have been exposed to more lead than people from rural areas.

3. Materials

Of the 29 individuals identified in the excavations, 13 had preserved teeth, and all of those had second molars present. All but one of these 13 came from pit F512 and only Sk6 from pit F514. For strontium, oxygen and lead isotope analyses, teeth were selected based on preservation, preferring those with less wear, and which were loose or easily extracted due to prior damage to the bone. They were fully recorded and photographed before sampling. Only Sk27A had extensive wear, all others had limited wear and no pathology. Using a dental burr, the surface enamel was abraded to remove any surface contamination. Approximately one third of the tooth was sectioned using a flexible diamond cutting disc and dentine was removed with a dental burr. The resulting clean, core enamel was crushed and spit into two aliquots for strontium and lead analysis and for oxygen isotope analysis.

4. Methods

a. Strontium and lead

Enamel samples were prepared following previously published procedures (Charlier et al. 2006; Smits et al. 2010). They were dissolved in 16M nitric acid, dried down and redissolved in 3M nitric acid. The samples were loaded on columns of Eichrom Sr-spec resin to separate Sr and Pb. Strontium was eluted with water and acidified to 3% nitric acid for analysis. Lead was eluted with 8N hydrochloric acid and diluted to 3N for analysis. All reagents were Teflon distilled and ultrapure Milli-Q system water was used.

Strontium isotope ratios were measured using a ThermoFinnigan Multi-collector ICP Mass Spectrometer (MC-ICP-MS) in the Northern Centre for Isotopic and Elemental Tracing at Durham University. Reproducibility of the standard NBS987 during analysis was 0.710258 ± 0.00011 (2σ , $n=12$). All values have been normalised to the accepted value of 0.710240 for NBS987.

Lead concentrations and isotopes were measured on the same mass spectrometer, using an Aridus desolvator nebuliser and x-skimmer cone to increase the sensitivity for the isotope measurements. Measurements of the standard NBS981 deviated from the accepted values (Baker et al. 2004) slightly more than measurement error, but insignificantly compared the variation being considered and have not been corrected. Lead concentrations have an uncertainty of approximately 10% of the measured value.

b. Oxygen

Enamel samples were prepared following previously published procedures (Smits et al. 2010) based on the rapid precipitation of silver phosphate (Dettman et al. 2001). Samples were dissolved using 2M hydrofluoric acid and 2M nitric acid to separate calcium as fluorite. The solution was neutralised and ammonia and silver nitrate added to precipitate fine-grained silver phosphate. After washing and freeze-drying, 0.15-0.20 mg of silver phosphate was weighed into silver capsules and submitted to the Laboratoire de Géologie de Lyon (CNRS-UMR 5276, Université Claude Bernard Lyon) for isotopic measurement in duplicate following the procedures of Fourel et al. (2011).

Replicate measurements on NBS120C prepared with the samples yielded a $\delta^{18}\text{O}$ value of 22.19 ± 0.63 ‰ VSMOW (1σ , $n=3$), and on NBS120C prepared in the Lyon laboratory by the method of Lécuyer et al. (1993) gave a value of 21.77 ± 0.37 ‰ VSMOW (1σ , $n=11$) which are both within error of the accepted value of 21.7 ‰ (summarized in Chenery et al. 2010). The technical error of measurement from all the duplicate samples in the batch was 0.35 ‰. Drinking water values ($\delta^{18}\text{O}_{\text{DW}}$) were derived from phosphate ($\delta^{18}\text{O}_{\text{P}}$) values using the calibration of equation 6 of Daux et al. (2008) for humans.

5. Results

Results for strontium and oxygen are tabulated in Table 1 and for lead concentrations and isotopes in Table 2.

Table 1: Samples analysed and results for strontium and oxygen isotopes. LL=lower left; LR=lower right , UL=upper left; UR=upper right.

Pit	Skeleton	Age	Sex	Tooth	$^{87}\text{Sr}/^{86}\text{Sr}$	$\pm 2\text{SE}$	$\delta^{18}\text{O}_p$ VSMOW	$\pm \text{sd}$	$\delta^{18}\text{O}_{\text{DW}}$ VSMOW
F512	Sk1	14-15½	—	ULM2	0.710702	0.000012	18.16	1.14	-5.76
F512	Sk2	18-25	M?	LLM2	0.709211	0.000018	18.69	0.89	-4.94
F512	Sk5	17-23	M	LLM2	0.708587	0.000014	15.56	0.81	-9.75
F514	Sk6	46+	M	LLM2	0.710141	0.000010	19.19	0.52	-4.17
F512	Sk12	19-23	M?	LRM2	0.709758	0.000012	failed		
F512	Sk19	18-25	M	LLM2	0.710037	0.000014	17.54	0.47	-6.70
F512	Sk21	21-25	M	LRM2	0.709967	0.000012	16.09	0.26	-8.94
F512	Sk22	18-25	M	URM2	0.713542	0.000016	19.11	0.34	-4.29
F512	Sk23	17-19	(M?)	LLM2	0.710592	0.000010	17.05	0.13	-7.46
F512	Sk24	17-18	(M?)	LLM2	0.708802	0.000012	14.71	0.11	-11.06
F512	Sk25	15½-16½	—	URM2	0.709760	0.000014	19.32	0.33	-3.97
F512	SK27A	36-45	M	LRM2	0.714684	0.000012	15.22	0.09	-10.28
F512	Sk28	16-20	M?	ULM2	0.709943	0.000012	19.17	0.32	-4.20

a. Strontium

The strontium isotope data is very varied, indicating that the Palace Green Library individuals could not have all spent their childhoods in the same locality.

b. Lead

Lead isotope ratios tend to be highly correlated and form linear arrays on simple plots of the measured ratios, as in Figure 2. An recently proposed alternative method of displaying archaeological lead isotope data is to transform the ratios by using a geological model for ore deposit formation and calculating the age of formation, and the ratios of uranium to lead (μ) and thorium to uranium (κ) before ore formation (Albarède et al. 2012). These parameters are independent of one another and thus sometimes give clearer plots without linear arrays of points. The data for the Palace Green Library individuals is plotted in both ways in Figures 3 and 4. One sample (Sk2) had very low lead levels and there was insufficient lead to make an isotopic measurement. Two samples (SK12, Sk25) plot away from the rest of the group, which is quite tightly clustered in the isotope measurements. The data show a wide variation in concentration, which is typical of past populations (Budd et al. 2004a).

c. Oxygen

Two of the samples encountered problems during the mass spectrometry. Sk1 has a larger than usual difference between the two measurements, but did not exhibit any other problems, so the mean of the two is used here, but should be regarded as relatively uncertain. The sample for Sk12 was contaminated with a carbon-containing material and neither measurement is reliable, so has no result. All the other samples pass quality control criteria for the measurement process. They are very varied and like the strontium results indicate that these individuals did not spend their childhoods in the same locality.

Table 2: Lead isotope ratio and lead concentration results

Skeleton	Pb /ppb	²⁰⁶ Pb/ ²⁰⁴ Pb	2SE	²⁰⁷ Pb/ ²⁰⁴ Pb	2SE	²⁰⁸ Pb/ ²⁰⁴ Pb	2SE	²⁰⁷ Pb/ ²⁰⁶ Pb	2SE	²⁰⁸ Pb/ ²⁰⁶ Pb	2SE	T	μ	κ
Sk1	165	18.45277	0.00095	15.62526	0.00103	38.4315	0.0036	0.846770	0.000020	2.082745	0.000104	207.67	9.6799	3.8878
Sk2	107	concentration too low to measure												
Sk5	257	18.44272	0.00092	15.62407	0.00102	38.4223	0.0033	0.847174	0.000017	2.083348	0.000091	213.37	9.6787	3.8890
Sk6	518	18.42591	0.00062	15.62337	0.00068	38.4169	0.0028	0.847909	0.000014	2.084913	0.000091	224.47	9.6791	3.8957
Sk12	588	18.28169	0.00068	15.58699	0.00075	38.2177	0.0029	0.852609	0.000012	2.090479	0.000088	283.18	9.6234	3.8785
Sk19	129	18.39871	0.00088	15.61614	0.00092	38.3790	0.0031	0.848758	0.000016	2.085972	0.000092	235.06	9.6678	3.8923
Sk21	216	18.38430	0.00075	15.61183	0.00085	38.3553	0.0033	0.849196	0.000015	2.086292	0.000090	240.10	9.6609	3.8887
Sk22	187	18.45954	0.00120	15.60964	0.00107	38.3674	0.0031	0.845615	0.000019	2.078491	0.000098	183.81	9.6485	3.8526
Sk23	445	18.38158	0.00127	15.61324	0.00125	38.3500	0.0037	0.849400	0.000015	2.086317	0.000084	243.74	9.6640	3.8882
Sk24	295	18.40472	0.00072	15.61664	0.00077	38.3783	0.0025	0.848513	0.000012	2.085251	0.000077	231.40	9.6682	3.8888
Sk25	714	18.20864	0.00069	15.55694	0.00073	38.1064	0.0031	0.854377	0.000012	2.092777	0.000104	299.18	9.5723	3.8623
SK27A	154	18.44800	0.00112	15.62093	0.00098	38.4178	0.0024	0.846762	0.000018	2.082550	0.000083	205.81	9.6719	3.8835
Sk28	156	18.38204	0.00085	15.61578	0.00083	38.3488	0.0028	0.849518	0.000015	2.086260	0.000076	246.47	9.6690	3.8880
NBS981														
	reference values	16.94160		15.49990		36.7258		0.914902		2.167788				
	measured	16.94175	0.00174	15.49856	0.00183	36.7171	0.0062	0.914807	0.000041	2.167250	0.000157			

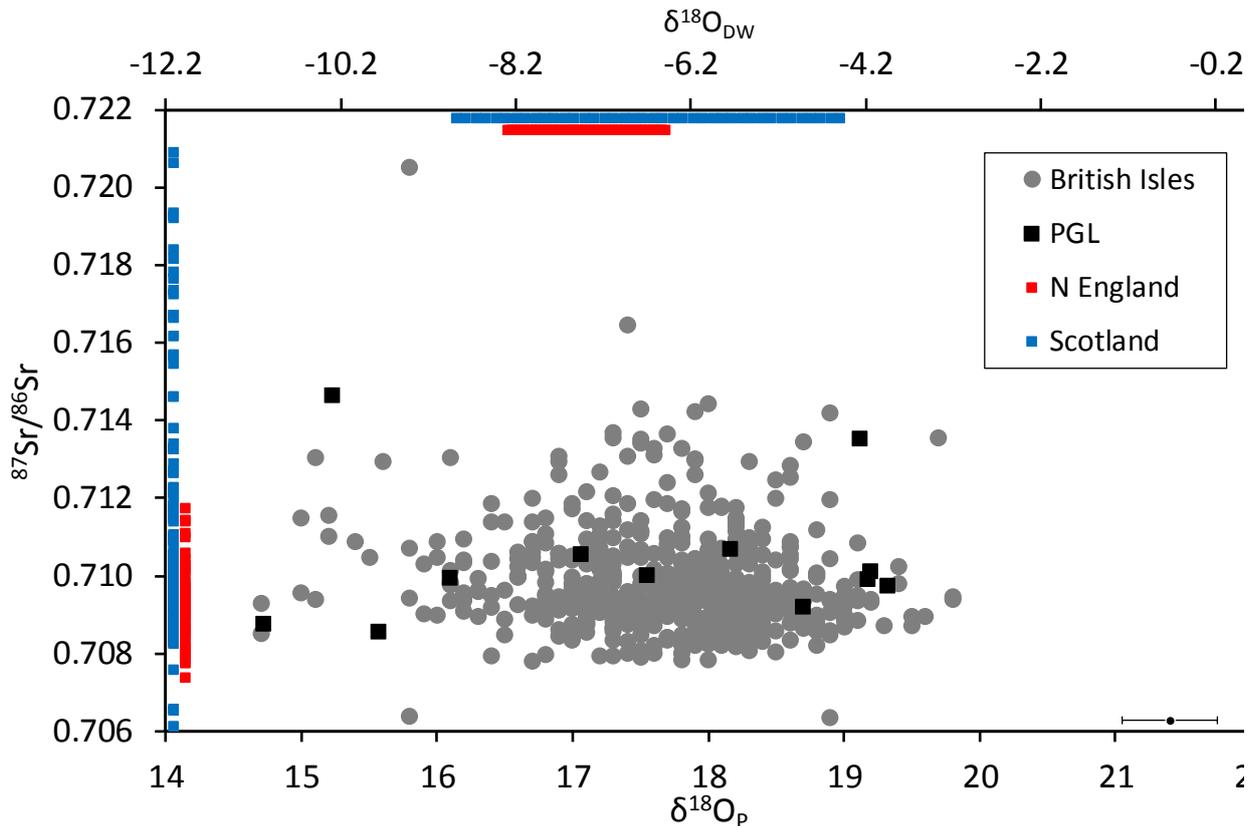


Figure 1: Plot of strontium and oxygen isotope data from Palace Green Library (black) overlaid on 584 published values for individuals buried in Great Britain (Evans et al. 2012). Bars at the top show indicative ranges of precipitation and groundwater $\delta^{18}\text{O}$ values for Scotland (blue) and northern England (red), based on (Darling et al. 2003). On the left are plotted strontium isotope ratios for Scotland (blue) and northern England (red) from the survey of environmental strontium isotope ratios by Evans et al. (2010). Error bars (TEM, 1σ) for the Palace Green Library $\delta^{18}\text{O}_p$ data are shown bottom right.

6. Discussion

Strontium and oxygen isotopes results are plotted in Figure 1 in comparison with a large dataset of values from Great Britain (Evans et al. 2012), and data indicating the ranges of these isotope ratio to be found in Scotland and northern England, here taken as north of 53.5°N , or approximately the mouth of the Mersey to the mouth of the Humber. The ranges of values for northern England are entirely encompassed within the ranges for Scotland, so any part of northern England cannot be distinguished from Scotland, but some parts of Scotland will be distinguishable from northern England. The environmental strontium isotope data represent a large dataset but do not sample every geological formation, so there may be more overlap than is indicated here.

In terms of strontium isotopes, two individuals (Sk22 and Sk27A) fall outside the observed northern England range, but within the Scottish range. Only four (Sk1, Sk19, Sk21 and Sk23) fall within the northern England $\delta^{18}\text{O}_{\text{DW}}$ range, or within measurement error of that range. Five fall to higher $\delta^{18}\text{O}_{\text{DW}}$ values within the Scottish range, but it should be noted that consumption of brewed or boiled liquids can raise $\delta^{18}\text{O}$ values by 2.3 ‰ or more (Brettell et al. 2012). Three individuals (Sk5, Sk24 and Sk27A) have lower values than are compatible with British drinking waters and such values are rarely found in human remains in Britain. These individuals are likely to be migrants from outside the British Isles. Such $\delta^{18}\text{O}_{\text{DW}}$ values are found in more easterly, cooler or

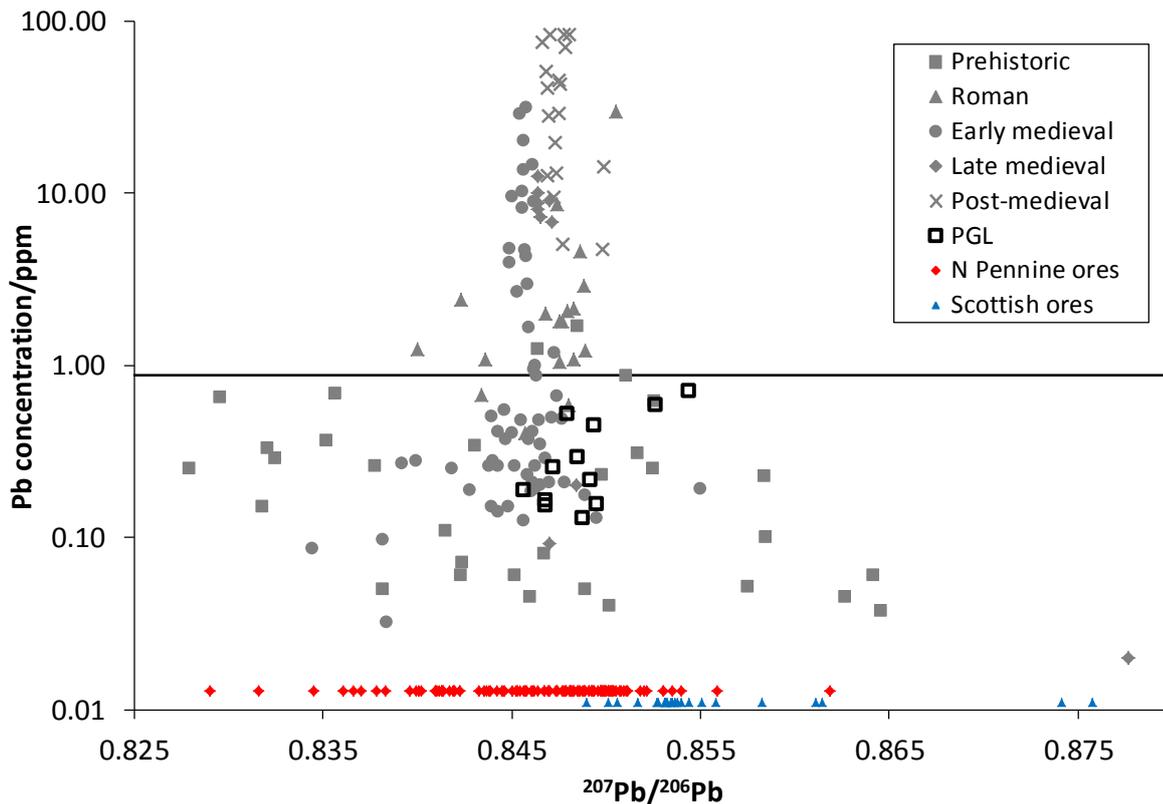


Figure 2: Lead concentration (logarithmic scale) versus $^{207}\text{Pb}/^{206}\text{Pb}$ ratios for the Palace Green Library individuals (black), plotted over the compilation of British data from Millard et al. (2014) (grey). At the bottom of the plot are values for Scottish (blue) and North Pennine (red) ore sources. The horizontal line represents the minimum at which anthropogenic exposure can confidently be identified according to Millard et al. (2014).

higher altitude environments within Europe (Bowen and Revenaugh 2003), such as Scandinavia, the Baltic or the Alps. Dutchmen (a term which then included Low Germans) and High Germans were recorded in the Scots army a few weeks after the Battle of Dunbar (Furgol 1990:316,329,342), but it is not clear if they were present at the battle itself, though some may have been (Furgol 1990:331). The strontium isotope values for these three individuals indicate multiple places of origin. More broadly, immigrant origins cannot be ruled out for almost any of the individuals as these combinations of strontium and oxygen isotope ratios can be found in many other places in Europe.

Figure 2 shows that lead concentrations in the Palace Green Library individuals are low compared to other post-medieval humans from England, but the others date from the 18th to 19th centuries, when lead use was significantly higher than in the 16th and 17th centuries. The Palace Green Library individuals all have concentrations below the limit of Millard et al. (2014) for unambiguously identifying anthropogenic lead exposure, though this does not mean that they were not exposed to anthropogenic lead. It is notable that two of the individuals with higher concentrations (Sk12 and Sk25) have isotope values similar to Scottish lead ores, and this also shows in Figures 3 and 4. Scottish lead-ore production was always small scale and additional ore was imported from England and the continent in the early modern period (Pickin 2010), thus some Scots may have an English ore signal. The isotopic range of environmental lead not from ore sources in the UK is broadly similar to the ore sources (Reimann et al. 2012), so it is only the combination of high concentrations and ratios close to those produced by cultural focussing

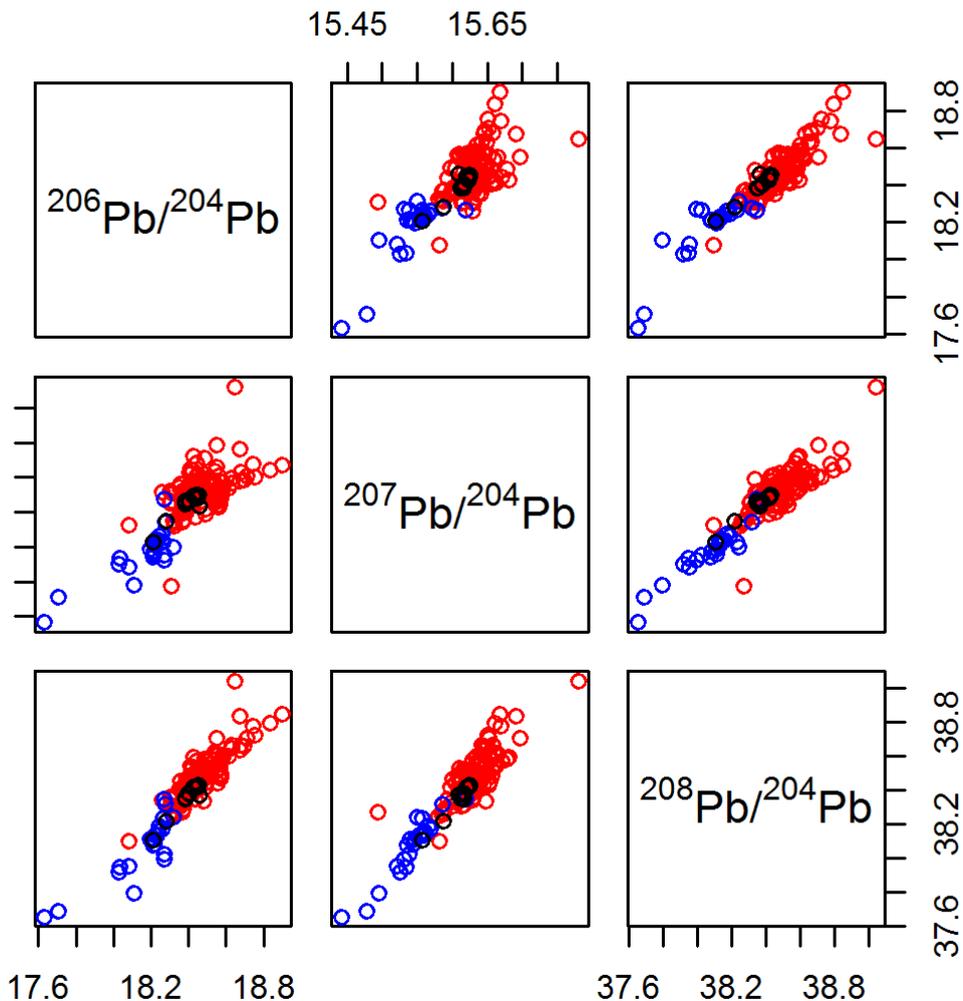


Figure 3: Lead isotope ratios for the Palace Green Library individuals (black) plotted over values from Scottish (blue) and northern Pennine (red) lead ores (Rohl 1996; Scaife et al. 2001; Shepherd et al. 2009) in all possible pairs of variables.

(Montgomery et al. 2010) that are diagnostic of location. From these data, therefore, SK12 and Sk25 may be tentatively identified as Scottish. The other individuals show ratios which may occur over much of England and Scotland, but their low concentrations suggest that they come from areas where the use of lead was restricted in the early 17th century. These are likely to be rural areas and poorer areas in towns, because those who could afford pewter adopted it quite widely from the late 16th century onwards, as Harrison (1587: 188) recorded that within living memory there had been “exchange of vessell, as of treene [i.e. wooden] platters into pewter”. The lead concentration and isotope data may thus be said to weakly favour a Scottish over an English origin, and a rural one over an urban one, but is not clear cut.

In Figure 5, other dimensions of the data are explored by plotting strontium isotope ratios and $\delta^{18}\text{O}$ values against lead concentrations and $^{207}\text{Pb}/^{206}\text{Pb}$ as a representative lead isotope ratio. The two individuals with high lead concentrations and distinctive lead isotope ratios (Sk12 and Sk25) can be seen to have identical strontium isotope ratios, and Sk25 has a high $\delta^{18}\text{O}$ value (Sk12 failed to yield good $\delta^{18}\text{O}$ data). Their compositions are consistent with (but do not prove) a common origin. The three individuals with low $\delta^{18}\text{O}$ values indicative of immigration do not show distinctive lead isotope ratios or concentrations.

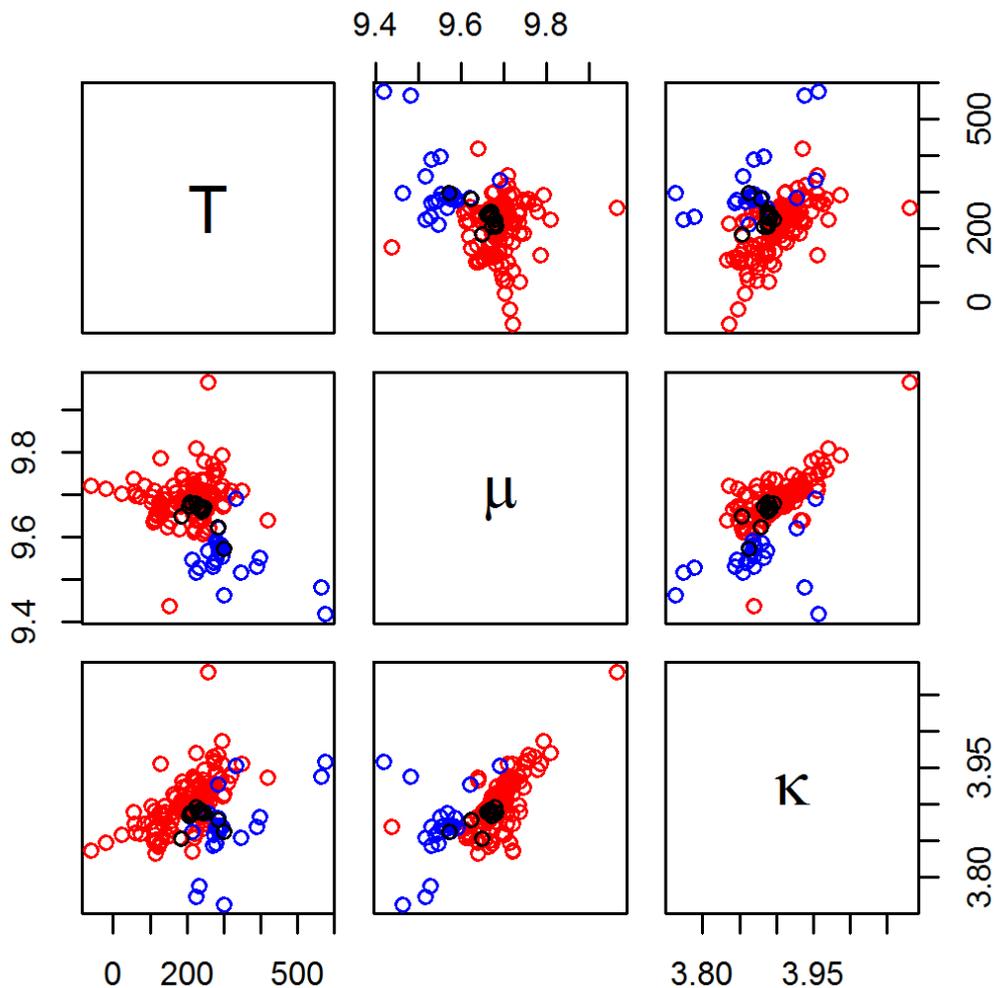


Figure 4: Lead isotope ratios of Figure 3 transformed to geological model ages (T), U/Pb (μ) and Th/U (κ) plotted in all possible pairs of variables. Colour key is the same as Figure 3.

Sk6 comes from a different pit to the other individuals, but does not stand out as particularly different, though he has the second highest $\delta^{18}\text{O}$ value and the third highest lead concentration.

7. Conclusions

The data show that whatever their origins, this group spent their childhoods in isotopically, and therefore geographically, diverse places. Four individuals, Sk1, Sk19, Sk21 and Sk23, are isotopically compatible with northern England or Scotland. Six individuals, Sk2, Sk6, Sk12, Sk25, Sk28, Sk22, are isotopically compatible with being from Scotland, although a boiling or brewing effect could place Sk2 to within the northern England range. Although Sk12 lacks the $\delta^{18}\text{O}$ data that excludes England for other individuals, his lead isotope ratios are more likely Scottish than northern English. Three individuals, Sk5, Sk24 and Sk27A, have $\delta^{18}\text{O}$ values indicating that they are likely to have immigrated into the British Isles since childhood.

In terms of the two hypotheses about the origins of the Palace Green Library individuals, the plague pit hypothesis would not predict that nine of thirteen were not from northern England. The Scots prisoners hypothesis would predict a majority compatible with origins in Scotland, and the possibility that mercenaries from Europe served in the Scots army could explain those with migrant origins.

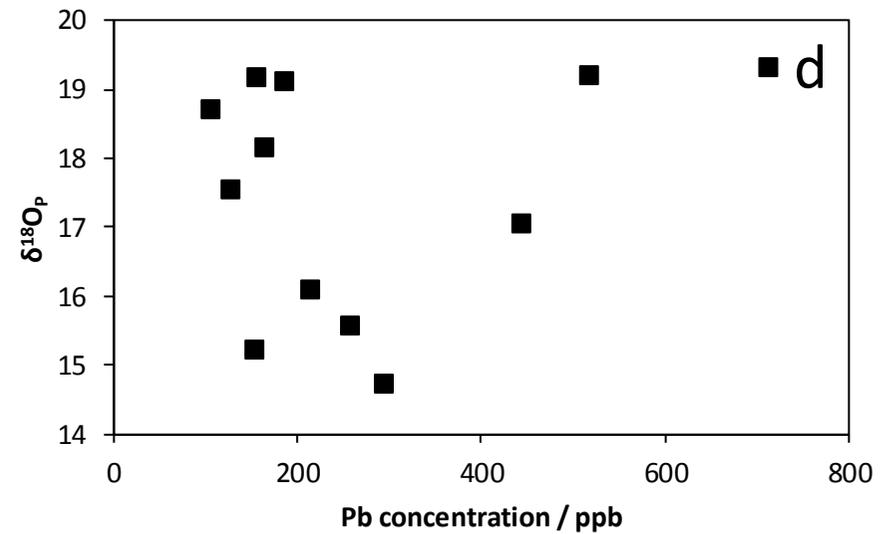
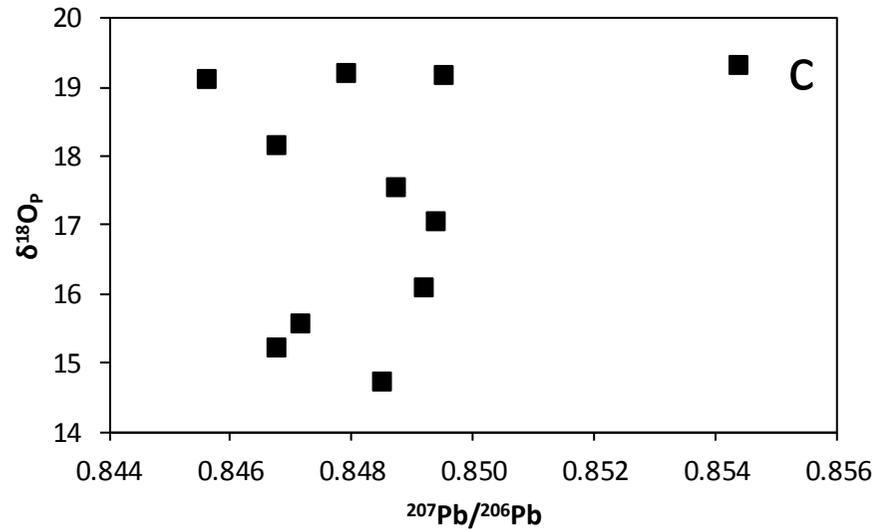
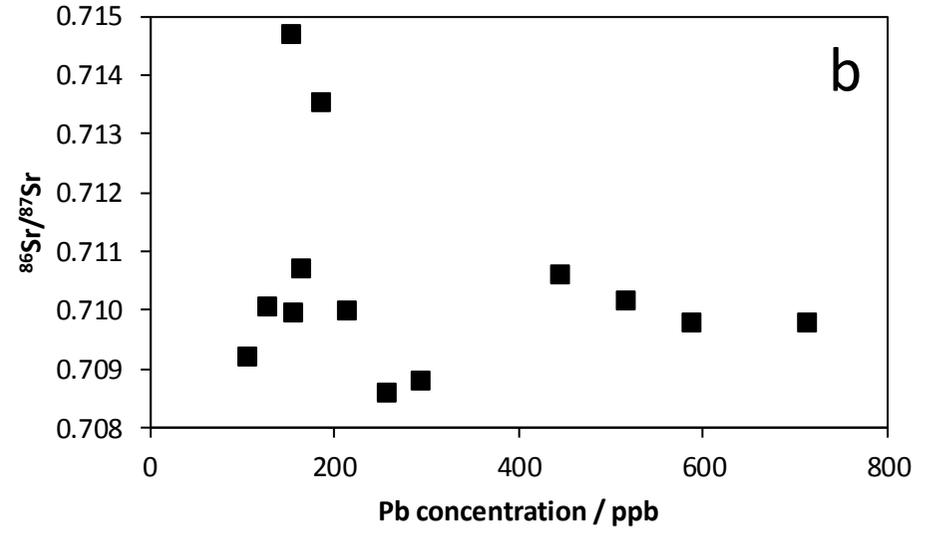
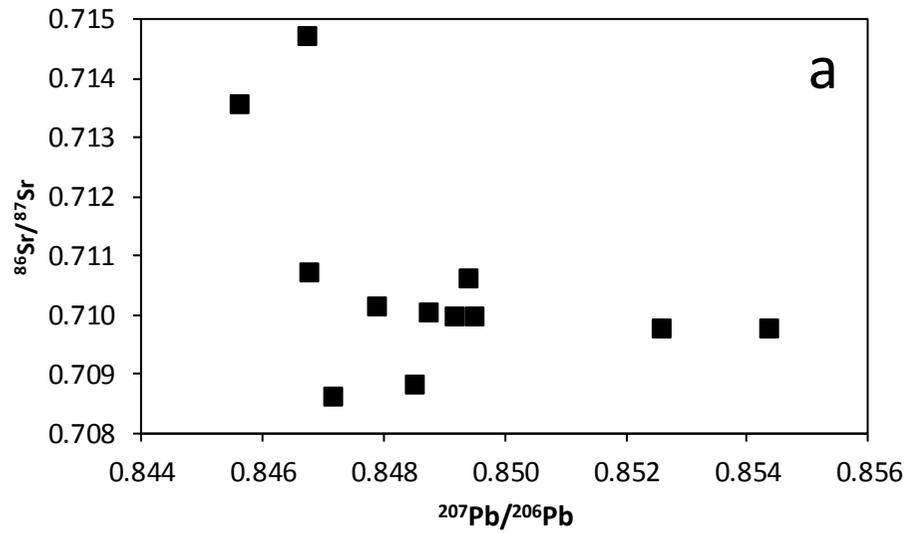


Figure 5: Measurement from the Palace Green Library individuals plotted as (a) $^{207}\text{Pb}/^{206}\text{Pb}$ ratio versus strontium isotope ratio, (b) lead concentration versus strontium isotope ratio, (c) $^{207}\text{Pb}/^{206}\text{Pb}$ ratio versus oxygen isotope value, (d) lead concentration versus oxygen isotope value.

Overall, it is more likely that as a group the Palace Green Library skeletons represent prisoners from the Battle of Dunbar than a group of people from Durham or northern England.

8. Acknowledgements

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