# A lithostratigraphical and chronological study of Oligocene—Miocene sequences on eastern King George Island, South Shetland Islands (Antarctica) and correlation of glacial episodes with global isotope events

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## Supplementary Information file S2 – full analytical details, spectra and isotope correlation diagrams for all new dated samples

#### <sup>40</sup>Ar/<sup>39</sup>Ar analytical methods

The argon isotopic results are listed in Tables S2-1 and S2-2. Age spectra and inverse isochron plots are shown in Figure S2-1. A summary of the <sup>40</sup>Ar/<sup>39</sup>Ar ages yielded in this study is provided in Table 4 (main paper).

#### New Mexico Geochronology Research Laboratory:

**Sample preparation and irradiation:** Hornblende separates and groundmass concentrates were prepared using standard mineral separation techniques (crushing, sieving, franzing and hand-picking). Samples were packaged and irradiated in machined Al discs for 7 hours in D-3 position, at the Nuclear Science Center, College Station, Texas. Neutron flux monitor was Fish Canyon Tuff sanidine (FC-1). Assigned age = 28.201 Ma (Kuiper et al., 2008).

**Instrumentation:** Mass Analyzer Products 215-50 mass spectrometer on line with automated all-metal extraction system. Samples step-heated in Mo double-vacuum resistance furnace. Heating duration 7 minutes. Reactive gases removed by reaction with 3 SAES GP-50 getters, 2 operated at ~450°C and 1 at 20°C, together with a W filament operated at ~2000°C.

**Analytical parameters:** Electron multiplier sensitivity averaged 1x10-16 moles/pA. Total system blank and background for the furnace averaged 350, 4.2, 0.6, 1.5, 1.9 x 10-18 moles at masses 40, 39, 38, 37, and 36, respectively for temperatures < 1300°C. J-factors determined to a precision of  $\pm$  0.1% by CO<sub>2</sub> laser-fusion of 4 single crystals from each of 4 radial positions around the irradiation tray. Correction factors for interfering nuclear reactions were determined using K-glass and CaF<sub>2</sub> and are as follows:

Texas:  $({}^{40}\text{Ar}/{}^{39}\text{Ar})\text{K} = 0.0002\pm0.0003$ ;  $({}^{36}\text{Ar}/{}^{37}\text{Ar})\text{Ca} = 0.00026\pm0.00002$ ; and  $({}^{39}\text{Ar}/{}^{37}\text{Ar})\text{Ca} = 0.00070\pm0.00005$ .

**Age calculations:** Total gas ages and errors calculated by weighting individual steps by the fraction of <sup>39</sup>Ar released. Weighted-mean ages were calculated using data from all heating steps (complete spectrum) from each of the groundmass concentrate samples by weighting by the inverse of the variance (Taylor, 1982), with weighted mean errors multiplied by the square root of the MSWD where MSWD exceeded cutoff values recommended by Mahon (1996). MSWD values were calculated for n-1 degrees of freedom for plateau and preferred ages. Isochron ages, <sup>40</sup>Ar/<sup>36</sup>Ar<sub>i</sub> and MSWD values calculated from regression results obtained by the methods of York (1969). Decay constants and isotopic abundances are after Steiger and Jäger (1977). All errors reported at  $\pm 2\sigma$  unless otherwise noted.

**Results:** Groundmass and hornblende were analyzed at by resistance-furnace incrementalheating <sup>40</sup>Ar/<sup>39</sup>Ar methods. Table 1 (main text) summarizes weighted mean ages and other results. Isochron results are listed in Table S2-1, and analytical data are detailed in Table S2-2. Figures showing age spectra and isochrons for individual samples are also provided.

All of the groundmass concentrates yielded somewhat non-flat age spectra with declining ages at highest temperatures, in some cases accompanied by older ages at low temperatures. Precision of individual steps varies markedly from sample to sample, which is at least in part a function of radiogenic yield (% Rad in Table 4 (main paper)). Sample P.2958.22 is an example of a low-yield, low-precision groundmass analysis, whereas P.2766.5 is an example of a higher yield, higher precision analysis in which the non-flat shape of the spectrum is more easily discerned. Accompanying K/Ca plots show that K/Ca ratios of the groundmass concentrates decline throughout the analysis of that sample; this is typical for basaltic groundmass, and represents lower temperature degassing of relatively K-rich plagioclase (±glass) followed by higher temperature degassing of K-poor pyroxene. As further discussed below, the shape of the spectra technically satisfy age plateau criteria (e.g. Fleck et al., 1977), it was not considered appropriate to calculate plateau ages for these flat segments within these recoil-affected spectra. Weighted mean Oligocene ages range from 24.09 ± 0.31 Ma to 27.56 ± 0.66 Ma, with a single Early Miocene age of 21.25 ± 3.14 Ma.

Two of the hornblende samples yielded relatively flat but somewhat imprecise age spectra, with low MSWD values (1.0 and 1.5) and weighted mean ages of  $24.09 \pm 0.31$  Ma (P.2800.1, MSWD=1.5) and  $26.80 \pm 0.69$  Ma (P.2767.11, MSWD=1.0). The third hornblende (P.2903.12 from Cape Melville) yielded a somewhat disturbed spectrum (MSWD=4.4) with a weighted mean age of  $21.25 \pm 3.14$  Ma, significantly younger than all other hornblende or groundmass samples.

All the data were also plotted on isochrons (Table S2-1, and see Figure S2-1, below) in an attempt to assess the possibility of initial trapped argon components with elevated  $^{40}$ Ar/ $^{36}$ Ar ratios indicative of excess  $^{40}$ Ar. Isochrons for hornblende analyses yielded moderately well-defined isochrons (MSWD = 1.0 to 4.4) with near atmospheric  $^{40}$ Ar/ $^{36}$ Ar intercepts and isochron ages in agreement with the weighted mean ages of their respective age spectra.

Isochrons for all fourteen of the groundmass samples are poorly defined with high MSWD values (2.8 to 38.9). In spite of this, intercept ages are all within  $\pm 2\sigma$  of their respective weighted mean ages (Table S2-1). Some of the <sup>40</sup>Ar/<sup>36</sup>Ar intercept differ by more than  $\pm 2\sigma$ 

of the atmospheric value of 295.5, but this is probably only an artifact of the <sup>39</sup>Ar recoil , which is also probably responsible for the disturbed shapes of the groundmass age spectra.

#### Leeds University:

The samples were crushed and sieved for the 250-500  $\mu$ m fraction, avoiding veining and weathered surfaces. The fractions were passed through a Frantz magnetic separator to concentrate plagioclase feldspar, washed in 1M HNO<sub>3</sub> for 10 minutes to remove carbonates, decanted then washed in 40% HF for a further 10 minutes to remove fines, rinsed in deionised water and then dried. The samples, weighing approximately 60 mg, were then hand-picked to remove visibly altered grains and grains with inclusions. The <sup>40</sup>Ar/<sup>39</sup>Ar analysis was carried out at the School of Earth Sciences, University of Leeds and followed the method described by Rex et al. (1993) with the following variations: Samples P. 2789.1 and P. 2792.4 were irradiated at the Riso Reactor, Roskilde Laboratory, Denmark, interference correction factors were (40/39)K = 0.048, (36/39)Ca = 0.38 and (37/39)Ca =1492. P. 2799.1 was irradiated at the McMasters Reactor, Ontario, Canada, interference correction factors were (36/39)Ca = 0.32, (37/39)Ca = 1515 and (40/39)K = 0.02. The University of Leeds internal standard is Tinto biotite (Rex and Guise, 1995) with assigned age of 409.2 Ma and biotite LP-6 (128.9 ± 1.4 Ma; Ingamells and Engels, 1976). Isotopic analyses were performed with a modified MS10 mass spectrometer, measured atmospheric <sup>40</sup>Ar/<sup>36</sup>Ar was  $287.8 \pm 0.2$  and sensitivity  $1.12 \times 10^{-7}$  cm<sup>3</sup>V<sup>-1</sup>. Gas volumes are corrected to STP.

The <sup>40</sup>Ar/<sup>39</sup>Ar ratio, age and errors for each gas fraction were calculated using formulae similar to those given by Dalrymple and Lanphere (1971). Errors in these ratios were evaluated by numerical differentiation of the equation used to determine the isotope ratios and quadratically propagating the errors in the measured ratios. J-value uncertainty is included in the errors quoted on the total gas ages but the individual step ages have analytical errors only. All errors are quoted at the  $2\sigma$  level unless otherwise stated. Ages calculated using the constants recommended by Steiger and Jäger (1977). Data for isotope correlation plots were reduced using the Isoplot/Ex program of Ludwig (1999). IsoPlot/Ex uses three separate models to regress the <sup>40</sup>Ar/<sup>39</sup>Ar versus <sup>36</sup>Ar/<sup>40</sup>Ar data on the isotope correlation plot. If the probability of fit of the initial regression is low, Isoplot attempts to use either a second or third model fit which weight the data-points using different criteria.

Potassium concentrations were measured using a Ciba-Corning 480 flame photometer incorporating a lithium internal standard. International and laboratory standards were analysed on a routine basis.

Argon was extracted in a glass vacuum line using a <sup>38</sup>Ar tracer from an aliquoting system. Special attention was given to the purity of the gas sample before it was analysed. A twostage clean-up procedure was used, stage one incorporating a Ti sponge furnace and liquid nitrogen trap. The gas was then transferred to a second stage Ti/Zr sponge furnace by absorption on activated charcoal at liquid nitrogen temperature. Argon isotopes were measured on a modified AEI MS 10 mass spectrometer fitted with computer controlled peak switching. Ion beams were detected by a VG pre-amplifier with 10<sup>11</sup> ohm resistor, digitized with a Solartron 7060 voltmeter and stored on computer disc for subsequent processing. International standards were analysed and atmospheric argon ratios determined on a regular basis. Ages were calculated using the decay constants and branching ratio agreed by the USGS Subcommission on Geochronology (Steiger and Jäger 1977).

#### Discussion

The age spectra of the hornblende samples are relatively simple, and the calculated weighted mean ages probably represent eruption ages. The relatively poor precision of the weighted-mean ages of hornblende separates probably reflect low K content coupled with low radiogenic yields related to alteration to chlorite and/or clay. Interpretation of results from the groundmass concentrates is less straightforward. The shape of the age spectra of the groundmass samples is almost certainly related to <sup>39</sup>Ar recoil, an experimental artefact of the neutron irradiation required for the <sup>40</sup>Ar/<sup>39</sup>Ar method (e.g. Lo and Onstott, 1989). Thus, during irradiation, <sup>39</sup>Ar is redistributed from K-rich to K-poor fine-grained phases, elevating the apparent ages of high-K, low-degassing temperature parts of the age spectrum, and depressing ages of high-K parts of the age spectrum, primarily highdegassing-temperature degassing pyroxene, but in some cases also probably including low-K, low-degassing-temperature alteration phases. In addition to <sup>39</sup>Ar recoil, the age spectra of groundmass concentrates may also reflect some disturbances related to alteration induced <sup>40</sup>Ar or K loss , and/or minor contributions of excess <sup>40</sup>Ar . No clear evidence of significant excess <sup>40</sup>Ar was observed, although this was considered to be a potential problem when this study was undertaken.

Calculation of accurate ages and uncertainties from sample age spectra that have suffered significant <sup>39</sup>Ar recoil is problematic. All steps for each sample were used to calculate the weighted-mean ages given in Table 4 (main text). The weighted-mean uncertainties values were calculated using the standard Taylor error, multiplied by the root of the MSWD in cases where the MSWD failed to meet the criteria of Mahon (1996). This conservative approach almost certainly results in overestimation of uncertainties. Analyses of one pair of samples from the same outcrop (Samples 2797.6A and 2797.6B) yielded similar weighted mean ages (24.99  $\pm$  1.38 Ma and 25.25  $\pm$  1.75 Ma) which agree much more closely than might be expected from the conservatively determined  $\pm 2\sigma$  uncertainty values.

#### References

- Dalrymple, G.B. and Lanphere, M.A. 1971. <sup>40</sup>Ar/<sup>39</sup>Ar technique of K-Ar dating: A comparison with the conventional technique. Earth and Planetary Science Letters, 12, 300-308.
- Fleck, R.J., Sutter, J.F. and Elliot, D.H. 1977. Interpretation of discordant <sup>40</sup>Ar/<sup>39</sup>Ar age spectra of Mesozoic tholeiites from Antarctica. Geochim. Cosmochim. Acta, 41, 15-32.
- Ingamells, C. O. and Engels, J. C. 1976. Preparation, analysis, and sampling constants for a biotite. National Bureau of Standards, Special Publication, 422, 401-419.
- Kuiper K. F., Deino A., Hilgen F. J., Krijgsman W., Renne P. R. and Wijbrans J. R. 2008. Synchronizing rock clocks of Earth history. Science, 320, 500–504.
- Lo, C.-H. and Onstott, T.C. 1989. <sup>39</sup>Ar recoil artifacts in chloritized biotite. Geoch. Cosmochim. Acta, 53, 2967-2711.
- Ludwig, K.R. 1999. Using Isoplot/EX, Version 2, a Geochronological Toolkit for Microsoft Excel. Berkeley Geochronological Center Special Publication, 1a.

- Mahon, K.I. 1996. The New "York" regression: Application of an improved statistical method to geochemistry. International Geology Review, 38, 293-303.
- Rex, D.C., Guise, P.G. and Wartho, J.-A. 1993. Disturbed 40Ar-39Ar spectra from hornblendes: Thermal loss or contamination? Chemical Geology (Isotope Geoscience Section), 103, 271-281.
- Rex, D. C. and Guise, P. G. 1995. Evaluation of argon standards with special emphasis on time scale measurements. In: Odin, G. S. (ed.) Phanerozoic Time Scale. Bulletin of Liaison and Information of the IUGS Subcommission on Geochronology, 13, 21–23.
- Steiger, R.H. and Jäger, E. 1977. Subcommission on geochronology: Convention on the use of decay constants in geo- and cosmochronology. Earth and Planet. Sci. Lett., 36, 359-362.
- Taylor, J.R. 1982. An Introduction to error analysis: The study of uncertainties in physical measurements. University Science Books, Mill Valley, California, 270 pp.
- Turner, G., Miller, J.A. and Grasty, R.L. 1966. The thermal history of the Bruderheim meteorite: Earth Planet. Sci. Lett., 1, 155-157.
- York, D. 1969. Least squares fitting of a straight line with correlated errors. Earth and Planet. Sci. Lett., 5, 320-324.

**Table S2-1.** Summary of new <sup>40</sup>Ar/<sup>39</sup>Ar step heating data for samples analysed at the New Mexico Geochronology Research Laboratory. Individual analyses show analytical error only; mean age errors also include error in J and irradiation parameters. hbl = hornblende separate; wr = whole rock; n = number of heating steps used to calculate plateau age.

ID	Temp	<sup>40</sup> Ar/ <sup>39</sup> Ar	<sup>37</sup> Ar/ <sup>39</sup> Ar	<sup>36</sup> Ar/ <sup>39</sup> Ar	<sup>39</sup> Агк	K/Ca	<sup>40</sup> Ar*	<sup>39</sup> Ar	Age	±2s
	(°C)			(x 10 <sup>-3</sup> )	(x 10 <sup>-15</sup> m	ol)	(%)	(%)	(Ma)	(Ma)
P.290	<b>3.12,</b> C3::	115 <i>,</i> 3.6 m	g hbl, J=0.00	0815218±0.09%	%, D=1.0064	14±0.000	91, NM-	115,		
Lab#=	50694-01									
А	3	159.4	0.3299	173.6	0.001	1.5	67.8	0.3	154.37	128.96
В	8	432.7	6.280	1327.9	0.007	0.081	9.4	2.0	60.15	85.93
С	12	55.09	19.83	137.0	0.046	0.026	29.5	13.5	24.47	3.54
D	16	29.07	20.88	54.41	0.137	0.024	50.6	47.8	22.21	1.18
Е	20	26.12	21.99	49.21	0.095	0.023	51.3	71.4	20.24	1.67
F	25	31.29	21.86	71.63	0.063	0.023	38.1	87.2	18.03	2.52
G	30	33.68	21.26	82.63	0.029	0.024	32.7	94.6	16.66	5.41
Н	40	68.52	20.33	174.3	0.022	0.025	27.3	100.0	28.16	9.10
isochr	ron age								19.02	3.68
total gas age n=3			n=8						22.34	4.37
weighted-mean age			n=8	steps A-H					21.25	3.14

## P.2767.11, C2:115, 8.36 mg hbl, J=0.000815312±0.09%, D=1.00468±0.00093, NM-115, Lab#=50693-01 A 800 695.0 2.207 2269.5 0.055 0.23 3.5 4.7 36.26 45.90

0.009

0.42

17.4

5.5

90.42

1001.2

В

850

357.9

1.211

68.19

С	950	215.4	2.692	679.3	0.011	0.19	6.9	6.5	22.11	45.73
D	1020	163.5	3.069	511.7	0.016	0.17	7.7	7.8	18.67	28.48
E	1080	147.2	6.915	412.5	0.024	0.074	17.6	9.9	38.33	19.87
F	1120	52.04	16.28	114.1	0.109	0.031	37.8	19.2	29.48	3.46
G	1160	33.84	19.27	60.60	0.588	0.026	51.8	69.8	26.38	0.83
Н	1200	30.35	17.88	45.88	0.118	0.029	60.2	79.9	27.45	2.23
I	1300	33.33	19.27	56.77	0.203	0.026	54.5	97.3	27.31	1.65
J	1400	133.6	157.0	435.3	0.010	0.003	13.4	98.2	30.82	36.48
К	1650	309.4	38.76	961.1	0.021	0.013	9.2	100.0	43.64	32.81
isochr	on age								26.03	1.04
total g	gas age		n=11		1.16	0.044			28.36	6.12
weigh	ited-mear	n age	n=11	steps A-K	1.16	0.044	46.4	100.0	26.80	0.69

**P.2766.5,** A5:115, 23.77 mg wr, J=0.000808835±0.09%, D=1.00468±0.00093, NM-115, Lab#=50685-01

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weig	hted-mean	age	n=9	steps A-I					25.23	0.97
total	gas age		n=9						24.72	0.40
isoch	ron age								25.71	0.36
I	1650	52.47	7.114	126.2	2.28	0.072	30.1	100.0	23.32	0.69
Н	1250	28.15	1.944	39.93	4.56	0.26	58.6	89.1	24.28	0.25
G	1075	21.85	0.9934	16.29	3.81	0.51	78.3	67.2	25.15	0.22
F	975	20.10	1.046	8.875	4.75	0.49	87.4	49.0	25.80	0.17
Е	875	20.98	1.173	12.66	2.76	0.43	82.6	26.3	25.47	0.21
D	800	46.52	1.098	100.6	2.05	0.46	36.3	13.1	24.79	0.63
С	750	63.59	1.126	161.8	0.179	0.45	25.0	3.2	23.35	2.55
В	700	101.8	1.119	301.1	0.313	0.46	12.7	2.4	18.99	2.76
А	625	121.0	1.022	374.2	0.186	0.50	8.7	0.9	15.54	4.13
Lubii	30003 01									

## **P.2958.22,** A1:115, 22.28 mg wr, J=0.000806369±0.09%, D=1.00468±0.00093, NM-115, Lab#=50681-01

	00002 02									
В	700	3200.4	2.159	10549.6	0.251	0.24	2.6	4.5	118.88	280.70
С	750	617.2	2.401	1925.5	0.028	0.21	7.8	5.0	70.20	114.03
D	800	399.1	3.565	1283.3	0.418	0.14	5.1	12.5	29.64	8.49
Е	875	338.1	7.083	1072.7	0.642	0.072	6.4	23.9	31.91	6.13
F	975	191.5	9.141	581.3	1.51	0.056	10.7	50.9	30.13	2.40
G	1075	52.36	6.613	115.2	0.718	0.077	36.0	63.7	27.75	1.28
Н	1250	58.45	5.668	138.6	1.24	0.090	30.7	85.9	26.43	0.88
I	1650	38.43	33.76	85.26	0.789	0.015	41.7	100.0	24.20	1.06
isoch	iron age								24.88	1.40
total	gas age		n=8		5.60	0.078			32.52	15.67
weig	hted-meai	n age	n=8	steps B-I	5.60	0.078	21.5	100.0	26.38	2.59

### **P.2960.22**, B3:115, 22.53 mg wr, J=0.00080965±0.09%, D=1.00468±0.00093, NM-115, Lab#=50689-01

Labi	50005 01									
В	700	1423.9	10.64	4765.2	0.041	0.048	1.2	1.7	24.71	154.58
С	750	1207.1	11.89	3682.5	0.005	0.043	9.9	1.9	170.99	521.07
D	800	816.3	19.68	2744.7	0.114	0.026	0.8	6.6	10.35	35.69
Е	875	362.6	19.41	1181.9	0.305	0.026	4.1	19.3	22.39	9.29
F	975	76.06	13.49	197.6	0.905	0.038	24.7	56.6	27.92	1.10
G	1075	47.32	10.48	100.5	0.489	0.049	39.1	76.8	27.41	1.07

Н	1250	51.42	22.95	115.5	0.285	0.022	37.3	88.6	28.76	1.65
I	1650	69.10	91.75	208.1	0.276	0.006	22.0	100.0	24.30	2.23
isochro	n age								28.25	1.81
total ga	is age		n=8						26.24	7.74
weighte	ed-mean	age	n=8	steps B-I					27.56	0.66

### **P.2007.2,** A3:115, 21.38 mg wr, J=0.000804986±0.09%, D=1.00468±0.00093, NM-115, Lab#=50683-01

LdD#-	-20002-01	-								
А	625	1967.7	3.485	6644.3	0.103	0.15	0.2	1.7	6.73	109.86
В	700	372.5	4.879	1218.6	0.132	0.10	3.4	3.9	18.84	13.81
С	750	192.9	4.545	607.3	0.031	0.11	7.2	4.4	20.37	18.43
D	800	216.5	5.923	666.7	0.362	0.086	9.2	10.4	29.30	4.38
Е	875	101.7	6.641	277.6	0.521	0.077	19.9	19.1	29.72	2.11
F	975	35.14	4.641	56.84	1.68	0.11	53.3	46.9	27.46	0.54
G	1075	25.37	3.957	24.93	1.40	0.13	72.2	70.1	26.86	0.44
Н	1250	27.91	6.723	36.68	1.29	0.076	63.2	91.6	25.90	0.52
I	1650	42.31	55.95	104.3	0.509	0.009	38.1	100.0	24.79	1.25
isoch	ron age								26.51	0.85
total gas age			n=9						26.49	3.20
weighted-mean age		n=9	steps A-I					26.73	1.19	

### **P.2962.1**, B2:115, 24.41mg wr, J=0.000809025±0.09%, D=1.00468±0.00093, NM-115, Lab#=50688-01

Lab#=	20688-01	<u>.</u>								
Α	625	3274.3	3.937	10967.9	0.102	0.13	1.0	2.2	49.21	225.96
В	700	388.7	3.980	1258.5	0.124	0.13	4.4	4.9	25.23	16.59
С	750	199.9	4.599	654.3	0.030	0.11	3.5	5.5	10.33	24.88
D	800	316.9	5.997	999.3	0.311	0.085	7.0	12.2	32.53	7.13
E	875	239.7	9.903	743.7	0.415	0.052	8.6	21.1	30.62	5.45
F	975	70.47	10.99	179.9	1.24	0.046	25.8	47.8	26.98	0.99
G	1075	31.10	7.357	47.26	1.05	0.069	57.1	70.4	26.22	0.49
Н	1250	39.73	10.74	80.47	0.773	0.048	42.4	87.0	24.96	0.71
I	1650	55.43	43.71	145.8	0.602	0.012	28.8	100.0	24.39	1.25
isochr	ron age								25.57	0.94
total g	gas age		n=9						27.17	7.22
weigh	ited-mea	n age	n=9	steps A-I					25.90	1.30

### **P.2774.10B,** B1:115, 22.24mg wr, J=0.000809495±0.09%, D=1.00468±0.00093, NM-115, Lab#=50687-01

20001-01	•								
700	1288.5	2.448	4279.9	0.132	0.21	1.9	1.2	35.20	70.00
750	865.0	1.811	2778.2	0.012	0.28	5.1	1.3	64.32	174.15
800	578.7	2.460	1875.3	0.856	0.21	4.3	9.0	36.34	7.96
875	137.3	2.624	396.9	1.32	0.19	14.8	20.9	29.84	1.76
975	47.44	3.079	103.1	3.71	0.17	36.3	54.1	25.36	0.53
1075	23.48	2.768	22.47	1.96	0.18	72.7	71.7	25.14	0.35
1250	22.76	3.615	21.49	1.44	0.14	73.4	84.6	24.63	0.35
1650	29.25	11.92	47.81	1.71	0.043	55.1	100.0	23.92	0.49
on age								24.48	0.53
total gas age		n=8						26.54	2.20
ted-mea	n age	n=8	steps B-I					24.88	1.20
	700 750 800 875 975 1075 1250 1650 ron age gas age ted-mean	700       1288.5         750       865.0         800       578.7         875       137.3         975       47.44         1075       23.48         1250       22.76         1650       29.25         ron age         gas age         ted-mean age	700       1288.5       2.448         750       865.0       1.811         800       578.7       2.460         875       137.3       2.624         975       47.44       3.079         1075       23.48       2.768         1250       22.76       3.615         1650       29.25       11.92         ron age       m=8         ted-mean age       n=8	700       1288.5       2.448       4279.9         750       865.0       1.811       2778.2         800       578.7       2.460       1875.3         875       137.3       2.624       396.9         975       47.44       3.079       103.1         1075       23.48       2.768       22.47         1250       22.76       3.615       21.49         1650       29.25       11.92       47.81         con age       gas age       n=8         ted-mean age       n=8       steps B-I	700       1288.5       2.448       4279.9       0.132         750       865.0       1.811       2778.2       0.012         800       578.7       2.460       1875.3       0.856         875       137.3       2.624       396.9       1.32         975       47.44       3.079       103.1       3.71         1075       23.48       2.768       22.47       1.96         1250       22.76       3.615       21.49       1.44         1650       29.25       11.92       47.81       1.71         ron age       gas age       n=8         rted-mean age       n=8       steps B-I	700       1288.5       2.448       4279.9       0.132       0.21         750       865.0       1.811       2778.2       0.012       0.28         800       578.7       2.460       1875.3       0.856       0.21         875       137.3       2.624       396.9       1.32       0.19         975       47.44       3.079       103.1       3.71       0.17         1075       23.48       2.768       22.47       1.96       0.18         1250       22.76       3.615       21.49       1.44       0.14         1650       29.25       11.92       47.81       1.71       0.043         con age       gas age       n=8         sted-mean age       n=8       steps B-I       5	700       1288.5       2.448       4279.9       0.132       0.21       1.9         750       865.0       1.811       2778.2       0.012       0.28       5.1         800       578.7       2.460       1875.3       0.856       0.21       4.3         875       137.3       2.624       396.9       1.32       0.19       14.8         975       47.44       3.079       103.1       3.71       0.17       36.3         1075       23.48       2.768       22.47       1.96       0.18       72.7         1250       22.76       3.615       21.49       1.44       0.14       73.4         1650       29.25       11.92       47.81       1.71       0.043       55.1         con age       gas age       n=8         red-mean age       n=8       steps B-I       5	700       1288.5       2.448       4279.9       0.132       0.21       1.9       1.2         750       865.0       1.811       2778.2       0.012       0.28       5.1       1.3         800       578.7       2.460       1875.3       0.856       0.21       4.3       9.0         875       137.3       2.624       396.9       1.32       0.19       14.8       20.9         975       47.44       3.079       103.1       3.71       0.17       36.3       54.1         1075       23.48       2.768       22.47       1.96       0.18       72.7       71.7         1250       22.76       3.615       21.49       1.44       0.14       73.4       84.6         1650       29.25       11.92       47.81       1.71       0.043       55.1       100.0         con age       gas age       n=8       steps B-I       steps B-I       164       1650 <th>700       1288.5       2.448       4279.9       0.132       0.21       1.9       1.2       35.20         750       865.0       1.811       2778.2       0.012       0.28       5.1       1.3       64.32         800       578.7       2.460       1875.3       0.856       0.21       4.3       9.0       36.34         875       137.3       2.624       396.9       1.32       0.19       14.8       20.9       29.84         975       47.44       3.079       103.1       3.71       0.17       36.3       54.1       25.36         1075       23.48       2.768       22.47       1.96       0.18       72.7       71.7       25.14         1250       22.76       3.615       21.49       1.44       0.14       73.4       84.6       24.63         1650       29.25       11.92       47.81       1.71       0.043       55.1       100.0       23.92         con age       24.48         gas age       n=8       steps B-I       24.88</th>	700       1288.5       2.448       4279.9       0.132       0.21       1.9       1.2       35.20         750       865.0       1.811       2778.2       0.012       0.28       5.1       1.3       64.32         800       578.7       2.460       1875.3       0.856       0.21       4.3       9.0       36.34         875       137.3       2.624       396.9       1.32       0.19       14.8       20.9       29.84         975       47.44       3.079       103.1       3.71       0.17       36.3       54.1       25.36         1075       23.48       2.768       22.47       1.96       0.18       72.7       71.7       25.14         1250       22.76       3.615       21.49       1.44       0.14       73.4       84.6       24.63         1650       29.25       11.92       47.81       1.71       0.043       55.1       100.0       23.92         con age       24.48         gas age       n=8       steps B-I       24.88

Lab#=	50686-01		-							
А	625	1412.0	2.552	4744.2	0.295	0.20	0.7	1.3	15.16	39.68
В	700	342.3	1.971	1106.3	0.492	0.26	4.5	3.4	22.86	6.81
С	750	99.97	1.886	269.4	0.097	0.27	20.5	3.8	30.11	5.28
D	800	77.73	1.927	201.5	2.11	0.26	23.6	12.9	26.94	0.99
E	875	31.35	2.322	48.47	3.39	0.22	54.9	27.5	25.32	0.33
F	975	19.63	2.064	7.408	5.52	0.25	89.7	51.2	25.89	0.13
G	1075	20.76	1.671	11.42	3.95	0.31	84.4	68.3	25.74	0.18
Н	1250	21.54	1.916	17.13	4.49	0.27	77.2	87.6	24.46	0.18
I	1650	30.30	8.114	48.82	2.89	0.063	54.6	100.0	24.47	0.37
isochr	on age								25.53	0.54
total g	as age		n=9						25.24	0.96
weighted-mean age n=9			n=9	steps A-I					25.42	0.88

**P.2780.1,** A6:115, 28.96 mg wr, J=0.000808474±0.09%, D=1.00468±0.00093, NM-115, Lab#=50686-01

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**P.2800.1,** C1:115, 19.35 mg hbl, J=0.000815613±0.09%, D=1.00468±0.00093, NM-115, Lab#=50692-01

Lau#-	-20032-01	•								
Α	800	1539.9	2.194	5109.0	0.027	0.23	2.0	1.0	44.88	135.69
В	850	427.0	1.713	1424.5	0.028	0.30	1.5	2.1	9.26	35.62
С	950	349.6	3.478	949.8	0.008	0.15	19.8	2.4	100.62	73.18
D	1020	179.8	6.339	544.1	0.020	0.080	10.9	3.2	29.03	23.32
Е	1080	91.25	13.21	266.9	0.030	0.039	14.8	4.3	20.21	9.86
F	1120	50.60	24.37	121.0	0.198	0.021	33.3	12.0	25.51	2.00
G	1160	25.16	23.45	37.09	1.32	0.022	64.1	62.7	24.40	0.41
Н	1200	19.89	15.10	18.18	0.489	0.034	79.3	81.5	23.67	0.62
I	1300	22.97	19.15	29.94	0.382	0.027	68.4	96.2	23.66	0.91
J	1400	62.18	29.47	172.5	0.069	0.017	21.9	98.8	20.77	4.33
К	1650	321.3	17.36	1059.2	0.030	0.029	3.0	100.0	14.63	32.03
isoch	ron age								23.98	0.54
total	gas age		n=11						24.30	3.43
weigł	nted-mea	n age	n=11	steps A-K					24.09	0.31
								-		

**P.2801.4,** A4:115, 19.41 mg wr, J=0.000807091±0.09%, D=1.00468±0.00093, NM-115, Lab#=50684-01

А	625	596.5	2.888	1971.5	0.069	0.18	2.4	0.6	20.85	38.75
В	700	78.36	2.210	204.6	0.187	0.23	23.1	2.2	26.56	2.80
С	750	35.16	2.148	58.24	0.074	0.24	51.6	2.8	26.59	3.76
D	800	31.87	2.159	48.10	1.20	0.24	56.0	13.2	26.17	0.53
E	875	20.36	2.438	11.50	2.02	0.21	84.3	30.6	25.19	0.27
F	975	18.01	2.159	3.672	3.41	0.24	95.0	59.9	25.11	0.16
G	1075	18.35	2.036	4.170	1.93	0.25	94.2	76.4	25.36	0.21
Н	1250	21.07	3.245	16.00	1.60	0.16	78.8	90.2	24.40	0.32
I	1650	34.43	11.80	67.47	1.15	0.043	44.9	100.0	22.92	0.57
isoch	ron age								25.19	0.40
total	gas age		n=9						24.97	0.58
weigl	hted-mean	n age	n=9	steps A-I					25.08	0.73

P.2797	<b>7.6A,</b> B4:1	115, 20.38 n	ng wr, J=0.000	810745±0.09	%, D=1.004	468±0.00	093, NM	-115,		
Lab#=	50690-01									
А	625	201.4	1.743	641.7	0.127	0.29	5.9	0.9	17.63	6.72

В	700	70.77	1.737	180.1	0.235	0.29	25.0	2.6	26.06	2.32
С	750	34.97	1.721	54.00	0.110	0.30	54.8	3.5	28.21	2.37
D	800	32.06	1.804	47.23	1.55	0.28	56.9	14.8	26.89	0.42
E	875	23.29	2.598	19.20	2.03	0.20	76.6	29.7	26.29	0.29
F	975	23.89	3.526	23.31	1.69	0.14	72.4	42.0	25.52	0.31
G	1075	28.07	2.935	38.22	1.40	0.17	60.6	52.3	25.11	0.41
Н	1250	32.03	2.095	53.74	4.53	0.24	51.0	85.4	24.07	0.32
I	1650	33.99	4.435	64.11	1.99	0.12	45.3	100.0	22.78	0.43
isochron age									26.29	1.49
total gas age n=9			n=9						24.82	0.46
weighted-mean age			n=9	steps A-I					25.25	1.75

**P.2797.6B**, A2:115, 21.67 mg wr, J=0.000804625±0.09%, D=1.00468±0.00093, NM-115, Lab#=50682-01

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Lab#-	-00002-01									
Α	625	64.23	1.147	179.7	0.224	0.44	17.5	1.3	16.43	3.00
В	700	29.70	1.070	44.24	0.582	0.48	56.3	4.8	24.43	0.95
С	750	22.90	0.9509	11.98	0.252	0.54	84.9	6.3	28.38	1.19
D	800	21.23	1.121	11.32	2.07	0.46	84.7	18.5	26.27	0.26
E	875	19.23	1.785	8.209	2.64	0.29	88.2	34.2	24.80	0.23
F	975	18.64	2.453	4.535	3.21	0.21	93.9	53.2	25.62	0.17
G	1075	20.59	2.342	13.70	1.99	0.22	81.3	65.0	24.50	0.26
Н	1250	26.17	2.250	34.51	3.93	0.23	61.7	88.3	23.66	0.24
I	1650	37.58	5.798	75.79	1.97	0.088	41.7	100.0	23.00	0.51
isochron age									25.66	0.59
total gas age			n=9						24.55	0.34
weighted-mean age			n=9	steps A-I					24.99	1.38

## **P.2797.10,** B5:115, 22.18 mg wr, J=0.000811215±0.09%, D=1.00468±0.00093, NM-115, Lab#=50691-01

Lab#-	-20021-01									
А	625	122.8	1.204	358.1	0.096	0.42	13.9	0.6	25.15	5.69
В	700	40.29	1.296	80.67	0.385	0.39	41.1	3.0	24.42	1.20
С	750	26.00	1.470	26.58	0.156	0.35	70.3	3.9	26.92	1.54
D	800	24.67	1.555	22.30	2.20	0.33	73.8	17.5	26.84	0.28
Е	875	20.11	1.995	9.365	2.74	0.26	87.1	34.3	25.82	0.21
F	975	20.67	2.297	12.33	2.86	0.22	83.3	51.9	25.40	0.19
G	1075	27.82	2.354	38.79	1.65	0.22	59.5	62.0	24.43	0.35
Н	1250	38.34	2.198	75.56	4.82	0.23	42.2	91.6	23.90	0.32
I	1650	51.38	7.195	127.9	1.36	0.071	27.6	100.0	21.04	0.66
isochron age								26.13	0.66	
total gas age n=9		n=9						24.75	0.37	
weighted-mean age		n=9	steps A-I					25.34	1.54	
								L		

**Table S2-2.** Summary of new  ${}^{40}$ Ar/ ${}^{39}$ Ar step heating data for samples analysed by Leeds University; J-value = 0.00592; n = number of heating steps used to calculate plateau age.

Sample	Temp	<sup>40</sup> Ar/ <sup>39</sup> Ar	<sup>37</sup> Ar/ <sup>39</sup> Aı	r <sup>36</sup> Ar/ <sup>39</sup> Aı	r <sup>39</sup> Ar <sub>κ</sub>	K/Ca	<sup>40</sup> Ar*	<sup>39</sup> Ar	Age	±2s
	(°C)			(x 10 <sup>-3</sup> )	) (x 10 <sup>-15</sup>	mol/g)	(%)	(%)	(Ma)	(Ma)
P2788.6	88.6 whole rock									
	535	0.63	9.20	8.60	282.8	0.054	99.1	3.7	9.8	16.1
	635	2.24	3.77	837.4	870.9	0.135	77.7	15.4	24.6	3.5
	700	2.60	2.50	279.7	814.4	0.200	90.7	26.4	27.9	4.5
	765	2.35	3.42	982.0	825.7	0.145	73.8	37.5	25.6	5.4
	820	) 2.82	4.78	1756.8	1017.9	0.105	66	51.2	29.9	2.5
	880	2.59	6.62	944.6	837.0	0.075	74.8	62.5	27.8	4.6
	950	2.23	7.18	1178.1	882.2	0.070	71.5	74.4	24.5	3.9
	1015	2.39	8.97	814.3	882.2	0.056	78.4	86.2	26.0	3.7
	1100	3.84	12.0	1056.9	667.3	0.042	67.9	95.2	39.1	6.1
	1285	5 13.1	50.0	321.7	350.6	0.010	78.5	100	121.1	8.8
total gas a	ge		n= 10						31.6	1.6
weighted-mean age n = 10		n = 10						27.0	5.0	
Plateau age (82.5% 39Ar; MSWD = 1.6)			) = 1.6)	Steps B-H					27.1	1.4

## Figure S2-1. Age spectra and isochron plots for King George Island samples



























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