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Archaeometallurgical residues from Llandre, Ceredigion

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Abstract

Iron production residues were recovered from two of the cut features excavated within the interior of the enclosure at Glanfred, Llandre. The assemblage include approximately 4.6kg of macro-residues derived mainly or entirely from iron smelting; small quantities of microresidues collected during the washing of the macroscopic specimens also included some hammerscale from ironworking (smithing).

The assemblage is dominated by flow slags with various surface textures and morphologies. The majority of the flow slags are simple elongate flows with free upper surfaces, but amalgamated blocks of flow slag and flows with contact with gravelly material on all surfaces also occur. The material is ambiguous as to the technology it represents: there are no moulds of wood or cereal pit-packing such as normally occur in non-tapping furnaces, how there is no surface – reddening of the flows, or amalgamation into large blocks of flowed slag as is normally seen in tapped furnaces. The assemblage most closely resembles unpublished material of similar age from Cefn Graianog, Gwynedd.

Two pieces of flow slag were selected for further detailed laboratory analysis. Bulk chemical analysis showed the slags were rich in manganese, but otherwise dominantly within the SiO_2-Al_2O_3-FeO system. Phosphorus was slightly elevated. Calcium, sodium and magnesium were all very low. Broadly similar elemental composition has been observed in slags from the smelting of bog iron ore in Gwynedd (Llwyn Du), Powys (Llandefaelog) and Pembrokeshire (Johnston, South Hook). The upper crust-normalised rare earth element profile was flat, probably indicating that the host sediment to the ore was fine-grained, or contained fine-grained bedrock.

In summary, the evidence suggests that the furnace was either non-tapping, or capable of tapping a low-volume of slag. It was smelting what was probably a local upland bog iron ore. Context (2003) probably presents mixed waste (either a primary dump) or as part of the workshop floor, whereas it is possible (but far from certain) that context (2005) might represent the highly degraded remains of furnace.

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Methods

The assemblage was visually inspected as part of an informal assessment. The catalogue is included as Table 1. Following the assessment, two samples were selected for further laboratory analysis.

The selected samples were slabbed on a diamond saw and subsamples were crushed for preparation of a whole-sample chemical analysis.

Bulk chemical analysis was undertaken using two techniques. The major elements (Si, Al, Fe, Mn, Mg, Ca, Na, K, Ti, and P) were determined by X-Ray Fluorescence using a fused bead on the Wavelength-Dispersive X-Ray Fluorescence (WD-XRF) system in the Department of Geology, Leicester University (this also generated analyses for S, V, Cr, Sr, Zr, Ba, Ni, Cu, Zn, Pb and Hf). Whole-specimen chemical analysis for thirty six minor and trace elements (Sc, V, Cr, Co, Ni, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Mo, Sn, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, Ta, Pb, Th, U) were undertaken using a sample in solution on the ThermoScientific ICAP-Qc quadrupole ICP mass spectrometer (ICP-MS) in the Department of Geology, Leicester University (this also generates lower quality results for Fe, Mn, Ti, P that are used mainly for QA purposes). The raw results of the chemical analyses are presented in full in the archive appendix (Appendix), with the key adjusted data presented as Tables 2 and 3. Adjustment has assumed all iron was originally present as FeO and all manganese as MnO. The assistance of Dr Tom Knott (XRF) and Dr Tiffany Barry (ICP-MS) is gratefully acknowledged.

This project was commissioned by Iestyn Jones.

The residues

Distribution of the residues

Residues were .recovered from the fills ((2003), (2005)) of two cut features of rather irregular plan. Context (2003) produced 4.54kg and context (2005) 105g of residues.

Context (2003) is described as the fill of irregular 'L'-shaped cut feature [2002], 2m (north to south) by 1.6m (east to west), shallow (0.13 to 0.2m deep), with rounded rides and a 'U' shaped base. The fill was a dark brown/black sandy-silt with charcoal inclusions, stones, furnace lining fragments and iron slag. A radiocarbon date obtained from charcoal associated with this deposit was dated to 1221+/-37 BP (UBA-24080), AD 688 to 889 (at 2 sigma).

Context (2005) was the fill of elongated (1.65m long, north to south amorphous; 0.8m wide) feature [2012]. A 0.5m wide slot through the fill of this feature revealed that it was also shallow (0.25m deep), with a flat base. The fill was similar in properties to fill (2003).

Neither feature was noted as containing evidence for in-situ heating, although photographs of [2002] do show possible reddening of the substrate close to the external side of its angle.

The features had been cut into a subsoil of shaley gravel, reflecting the underlying Silurian mudstones of the Borth Formation.

Description: iron smelting macro-residues

The macroscopic smelting residues were divided (Table 1) into several classes:

Flow slags with smooth surfaces: these flow slags show upper surfaces with smooth, dark surfaces and a shiny lustre. In this, they resemble tapped slags (i.e. slags which had been tapped so that they had flowed out of the furnace before cooling). Flow slags in non-tapping furnaces (i.e. those furnaces in which all the slags cooled within the furnace) may also show free surfaces, so this is not a firm discriminating factor. The smooth flow slags did not show any superficial reddening (which forms in tapped slags because of the superficial oxidation of the slag producing a thin layer of haematite), but this is not always a clear discriminant, because some high-manganese tapped slags also may not show much reddening.

These flow slags were mainly either in individual elongate flow lobes/tubes, or in small amalgamations. There were only a few pieces in which the individual prills were more numerous. None of these amounted to a substantial block and they were mostly just a single layer of flow lobes in thickness.

Flow slags with smooth surfaces comprised 1950g (124 pieces) of the assemblage (

Flow slags with gravelly surfaces: some of the flow slags showed dull surfaces with abundant fine gravel inclusions (12 pieces; 330g). Some of these were in well-formed flow lobes, but others were in the form of elongate, rod-like, bodies.

Slag rods are probably most commonly formed by slag entering holes pushed below the furnace charge by a tool (typically an iron rod). Such rods would be more likely to be formed in a slag-tapping furnace, during management of the tapping process. They could theoretically be generated during use of a rod to clear the hot slag from a non-tapping furnace too, but this is much less likely.

Hearth/furnace lining: the assemblage included 19 fragments (weighing 254g of furnace/hearth lining from (2003) and 3 pieces (weighing 10g) from (2005). The fragments were generally small and undiagnostic. No pieces showed evidence for the air supply.

Smithing hearth cake/furnace bottom: the assemblage contained a single large block (380g) containing what appears to be part of the margin of a plano-convex slag cake. The piece shows signs of having been deformed when hot and is difficult to orientate with certainty. It shows a small area of what is probably a smooth top, adjacent to an inclined, gravelly side.

Two other dense slag fragments (54g and 100g) were probably burr fragments (the zone of interaction between the hot slag and hearth/furnace wall just below the air inlet). One of these (a 100g fragment) might alternatively be a fragment of a smithing hearth cake.

Indeterminate slag: in addition to the above pieces (all from (2003) except where indicated), there were (from (2003)) 20 pieces (664g) of slag fragments showing some signs of flow. These were all

indeterminate in origin, but might potentially include fragments of flow slag accumulations (but lacking characteristic surfaces) and fragments of furnace bottoms. There were also 6 pieces or lumps of slag that were particularly 'rusty', suggesting that they might have contained metallic iron. Again, an origin as furnace slags (or furnace bottom) might be likely, but not evidenced by the morphology of the pieces. There were three fragments (18g) of tis rusty sheet-like material, possibly fragments of ferruginous weathering crusts, but an origin in the weathering of a thin iron sheet is also possible. Finally there were 40 pieces (468g) of iron slag fragments there were entirely indeterminate.

Context (2005) produced 6 pieces of indeterminate slag, all, low density, some in moderately large flow lobes and some possibly brecciated and perhaps related to the 'sinter' facies reported from the base of some non-tapping furnaces (e.g. Young 2008). These slags were mostly highly vesicular, pale below the dark surface, and coated in ashy deposits.

Description: iron smithing micro-residues

As no samples were available for the investigation of microresidues, all sediment removed during the washing of the macroscopic samples was collected and wet-sieved at 63µm, before magnetic separation.

For the material from context (2003), the washings were rich, not only in slag debris, but in flake hammerscale (Young 2011). This hammerscale was in small fragments of thin flakes.

The washings from context (2005) did not produce any hammerscale, despite being rich in slag debris and charcoal. These washings were extremely rich in very fine grained black material, probably secondary manganese oxides.

Chemical composition of residues

Bulk major element composition

The adjusted major elemental compositions of the analysed residues are provided in Tables 2 and 3.

The major element composition of these two samples may conveniently be considered within the system SiO_2-Al_2O_3-FeO (Figure 1; after Schairer and Yagi 1952, fig 6) because these three oxides together comprise a very high proportion of the total. The low concentrations of all the other 'major' elements is noteworthy. The analyses plot close to the fayalite-hercynite divide

When these analyses are recast on an iron-free basis the compositions of the analysed samples are remarkably similar (Table 4).

Trace elements

The trace elemental compositions of the residues are provided in Table 3. The contents of most trace elements in the slags are relatively low.

The rare earth elements (REE) show almost flat upper crust-normalised profiles (normalisation after Taylor & McLennan 1981; Figure 2).

Interpretation

The morphology of the slags suggests either that they formed in the basal pit/chamber of a non-tapping furnace, or that some of the slags were tapped in low volumes. The presence of gravel within some of the slags is in accordance with the very loose substrate into which the features were dug (as presumably were the features in which the slags originated).

Context (2003) contained both iron smelting macro-residues and smithing microresidues (hammerscale). Such deposits may develop on workshop floors, as well as accumulate in adjacent negative features. Many of the contexts at Cefn Graianog (Young 2015) contained mixed assemblages of this type. Context (2005), although only having a tiny amount of matrix available for examination, did not contain hammerscale. The slags from this context were indeterminate, but with a higher probability of presenting material in-situ in metallurgical feature because of the ashy nature of both matrix and slag; the lack of hammerscale would argue for any such primary feature being from smelting (as would, circumstantially, the elongate nature of the feature, which is of an appropriate size for the highly truncated remans of an early slag tapping furance).

The limited amount of data available for the assemblage means that a full interpretation of the chemical composition in terms of furnace mass balance is not possible. Nonetheless, the data may be compared with analyses of flow slags/tapped slags from other sites.

Chemical analytical data are available for several sites in Wales that have produced analyses interpreted as indicating the smelting of bog iron ores. These sites include Brownslade, South Hook, Steynton (ore only) and Johnston in Pembrokeshire, Llandefaeolog in Powys and Llwyn Du in Gwynedd. As well as being from geographically distinct areas, these sites also lie on differing bedrock geology (although it must be borne in mind that the ore did not necessarily derive from a locality with the same geology): Precambrian: Johnston, Cambrian-Ordovician: Llwyn Du, Silurian: Llandre, Silurian-Devonian ('Old Red Sandstone'): South Hook, Steynton, Landefalelog, Carboniferous: Brownslade.

The major element analysis of the Llandre slags is compared with those of the comparative sites in Figure 3.

The data indicate that the medieval smelting of bog iron ores developed on Cambrian-Ordovician bedrock in Gwynedd was markedly different from that of the earlier smelting of ores from areas of Precambrian and Old Red Sandstone geology in Central and West Wales. The major change in technology over this period complicates the interpretation (as does the different analytical technique that was applied to the Llwyn Du material, potentially providing less accurate values). However, the geological setting is interpreted as being one of the major controls in the differences. The geological setting would influence both the furnace construction materials and the nature of the ore. Of the major elements, only manganese and phosphorus are likely to be dominantly influenced by the ore, where the other major elements are likely to be most influenced by the composition of the furnace.

The Llandre slags plot as marginal to the Llwyn Du slags on all the diagrams, including those featuring manganese and phosphorus. They are of higher phosphorus content than the majority of the Llwyn Du

ores, but contain significantly less phosphorus, than any of the more southern examples. The manganese content of the Llandre slags is somewhat low compared with the typical content observed in the examples, but within their range of compositions. The Llandre slags are also intermediate between the Llwyn Du and South Hook slags in terms of the Mn:Ba ratio, but on this metric the Llandefaelog samples are differentiated from the Pembrokeshire samples, with much lower MnO/Ba driven by elevated levels of barium.

Comparison of the REE profiles is complicated by the poorly understood relative influence of the host sediment and the iron mineralisation on the REE. It currently seems likely that the REE profile is more strongly influenced by the host sediment. The profile for the Llandre slags is very flat and low (with just a very slight downwards inclination of the LREE), probably reflecting the influence of a fine-grained (mudrock; shale/slate) host sediment on the ore (and probably also on the furnace ceramic). A similar flat, low profile, was observed for samples from the Llandefaelog slags (they also show an apparent positive europium anomaly, but this may be a poorly-corrected spectral overlap with [BaO]+). In contrast the data from Pembrokeshire area show profiles with variable elevation of the MREE, reduced LREE and a negative cerium anomaly, reflecting a more complex host sediment, probably with a strong influence from the volcanic rocks of the Skomer Volcanic Group., and possibly also the influence of a coarser-grained host sediment

Summary

The analysis presented above suggests that the samples are bloomery smelting slags from the smelting of a bog iron ore, with a chemical composition quite similar to that of smelting slags (from Llwyn Du) from the smelting of the bog iron ores developed over mudrock bedrock on the eastern side of the Harlech Dome. There are, however, far too few examples of comparative material to produce any real predictive modelling of the characteristics of the source.

The presumed source for the Llwyn Du smelting operation are the upland blanket bogs of the Crawcwellt area (Crew 2009). It is likely that the ores smelted at Llandre were also from an upland bog. One surviving area of peat lies approximately 1km west of Llandre and there were probably other areas of impeded drainage before farmland improvement. It is also possible that there were iron ores associated with the margins of Cors Fochno - although there are no descriptions of iron enrichment in the lowland raised bogs of Wales known to the author (raised bogs, however, do appear to have provided a major resource of iron ore in early times in Ireland). The resource need not have even been in a true bog; deposits that may be termed bog iron ores also form where groundwater leaks (and oxidises) from an area with impeded drainage, in which reducing conditions have allowed the accumulation of iron in the groundwater from weathering of the bedrock.

The technology of the iron smelting is still uncertain. Clarification of this would be highly desirable as the early medieval period shows a complex variation of approaches to iron smelting with both time and geographical location.

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Figure Captions

Figure 1: major element composition of bulk analyses of residues plotted within the ternary system SiO_2-Al_2O_3-FeO (fields after Schairer and Yagi 1952, fig 6). Red circles: analyses of flow slags from Llandre. Black crosses: analyses of tapped slags from Llwyn Du (data from Charlton *et al*. 2010)

Figure 2: upper crust-normalised rare earth element (REE) profiles (normalisation after Taylor & McLennan 1981) for analyses of flow slags from Llandre.

Figure 3: Binary plots of oxide concentrations (in wt%) from bulk chemical analyses (by XRF and ICP-MS) Analyses of slags from Llwyn Du from Charlton *et al*. 2010; from Brownslade after Young 2010a; from South Hook after Young 2010b; from Johnstone and Llandefaelog after Young 2014; and those of the ore from Steynton after Young 2014.

Table 1: summary catalogue. SHC = smithing hearth cake, All weights in gram.

context	*Wt.*	*No.*	*notes*
2003	*1950*	*124*	*Flow slag with smooth surfaces with bright lustre; mostly fragments of isolated prills, but also includes amalgamated flows with multiple narrow prills, some forming in a 'V'-shaped trough – and this resembling tap slags, except having dark surfaces and only one prill layer in thickness. Some pieces show some gravel embedded in base.*
	330	*12*	*Flow slags with much embedded gravel and dull, rough, surfaces; many of these prills are rod-like.*
	254	*19*	*Vitrified hearth/furnace lining*
	380	*1*	*Fragment containing curved side of a slag bowl; difficult to orientate, probably hot-deformed; shows irregular gravelly basal contact and a small area of probable upper surface that is smooth; might be either a basal smelting slag or a deformed SHC*
	54	*1*	*Small fragment of burr*
	70	*1*	*Dimpled tool mould, fractured from a larger slag cake*
	100	*1*	*Dense fragment of either burr or SHC*
	664	*20*	*Broken slag fragments with some indications of flow, but not apparently flow slags*
	252	*6*	*Amorphous fragments/lumps of slag with rusty surfaces, probably contained metallic iron*
	18	*3*	*Very thin rusty sheets of iron oxide; probably contractionary, but just possibly after iron metal*
	468	*40*	*Indeterminate small slag fragments*
	84	*7*	*Natural stone fragments*
2005	*95*	*6*	*Blebby porous and slightly lobate iron slag; some almost of sufficiently low-density to be termed fuel ash slag*
	10	*3*	*Vitrified hearth/furnace lining*

Table 2: Major elements by XRF. < = below detection. All elements presented as wt% oxide. Adjusted to Mn^{2+} and Fe^{2+} and adjusted to exclude volatiles (LOI).

	SiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5
LLE1	24.52	8.35	55.53	6.87	0.37	0.77	0.40	1.23	0.38	0.33
LLE2	24.72	8.51	55.12	6.98	0.45	0.87	0.55	1.50	0.36	0.34

Table 3: trace elements by ICP-MS. < = below detection. All elements presented as ppm. Adjusted to Mn^{2+} and Fe^{2+} and adjusted to exclude volatiles (LOI).

	Co	Cu	Ga	Ge	Rb	Sr	Y	Zr	Nb	Mo	Ru	Sn	Cs	Ba	La	Ce	Pr
LLE1	21.65	3.01	10.02	17.32	27.95	85.27	11.64	84.96	10.56	3.68	0.01	0.19	1.50	518.14	9.89	27.04	2.80
LLE2	21.62	2.66	9.85	17.21	21.55	88.10	8.79	86.33	10.48	3.71	0.01	0.22	1.67	558.63	11.19	25.12	3.06

Table 3 (continued): Minor and trace elements by ICP-MS. < = below detection. All elements presented as ppm. Adjusted to Mn^{2+} and Fe^{2+} and adjusted to exclude volatiles (LOI).

	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Hf	Ta	W	Pb	Th	U
LLE1	12.49	2.79	0.63	2.56	0.36	2.16	0.45	1.31	0.19	1.44	0.18	2.14	0.42	0.59	1.65	3.23	1.08
LLE2	13.67	2.66	0.54	2.37	0.33	1.99	0.40	1.15	0.16	1.15	0.15	2.16	0.38	0.50	1.41	2.97	1.10

Table 4: Major elements by XRF as Table 2, normalised to exclude iron.

	SiO_2	Al_2O_3	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5
LLE1	56.43	0.87	19.21	15.80	0.84	1.78	0.93	2.83	0.76
LLE2	55.53	0.82	19.11	15.68	1.01	1.96	1.25	3.37	0.76

Figure 1

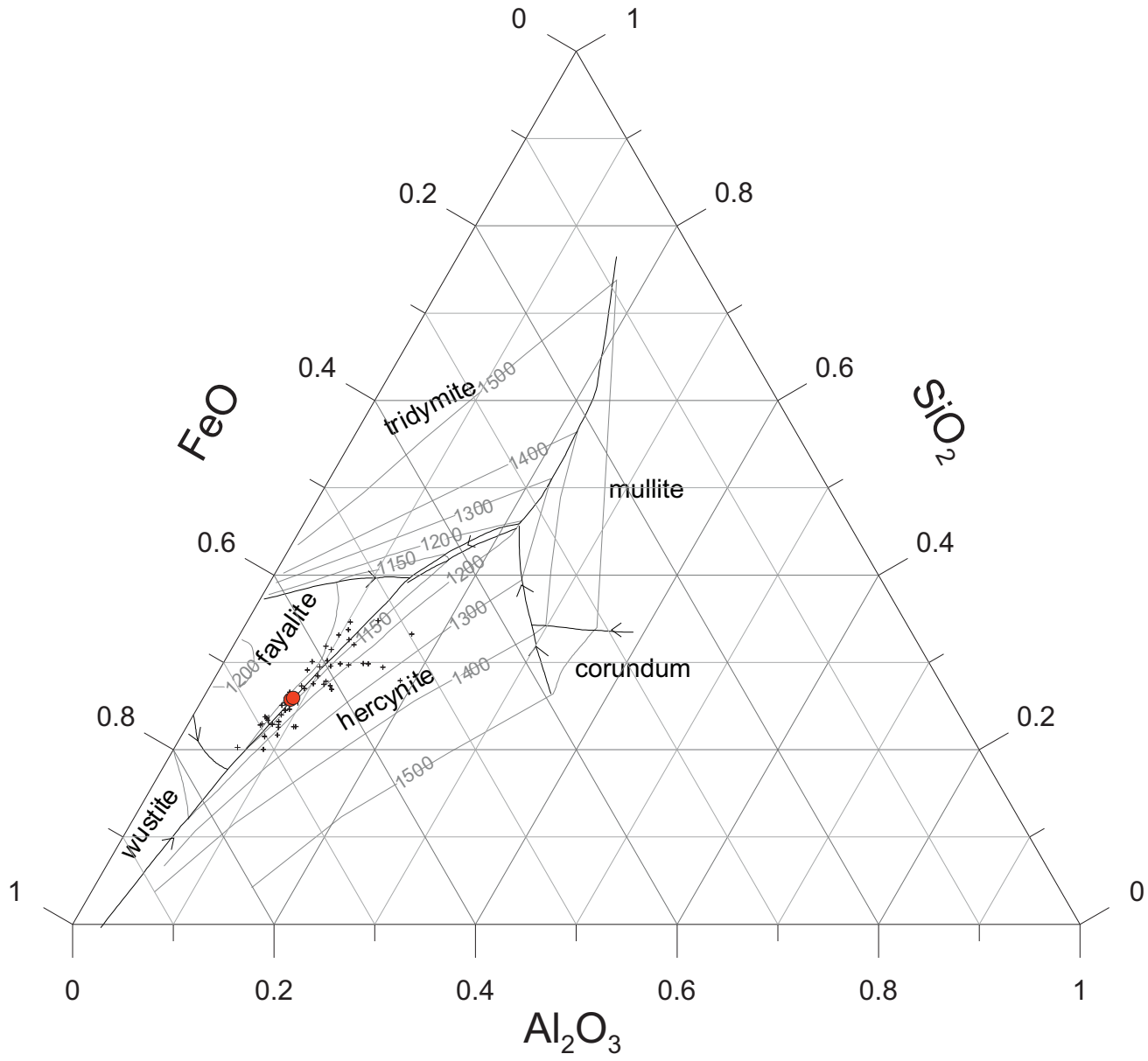
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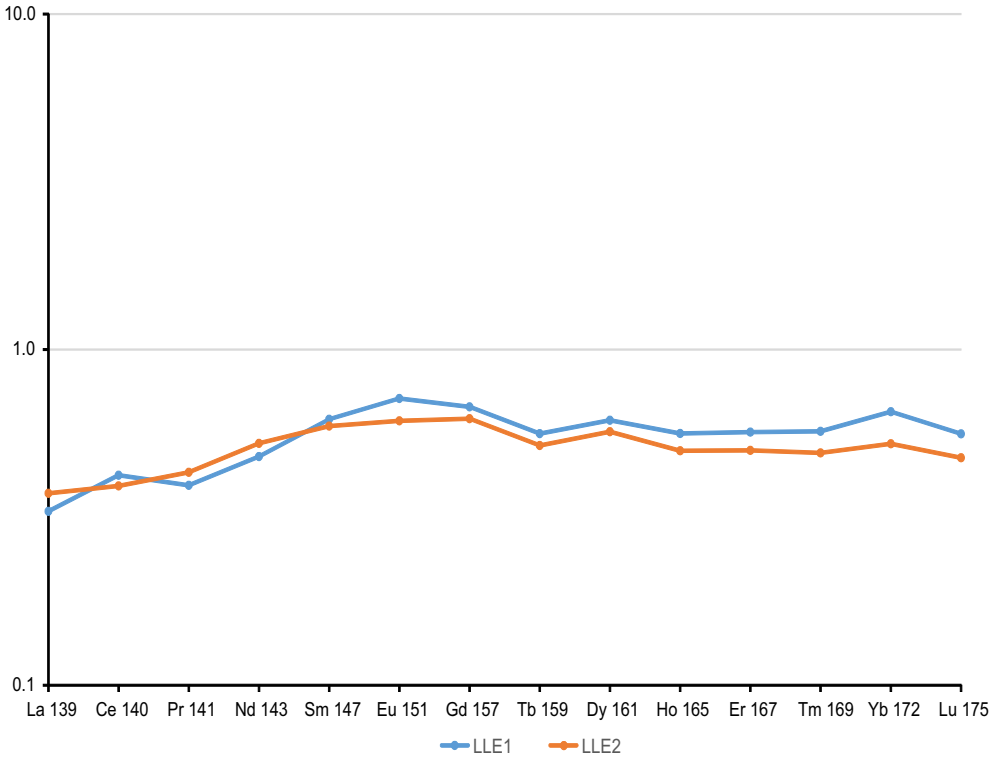
Figure 2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Ce 140
Pr 141
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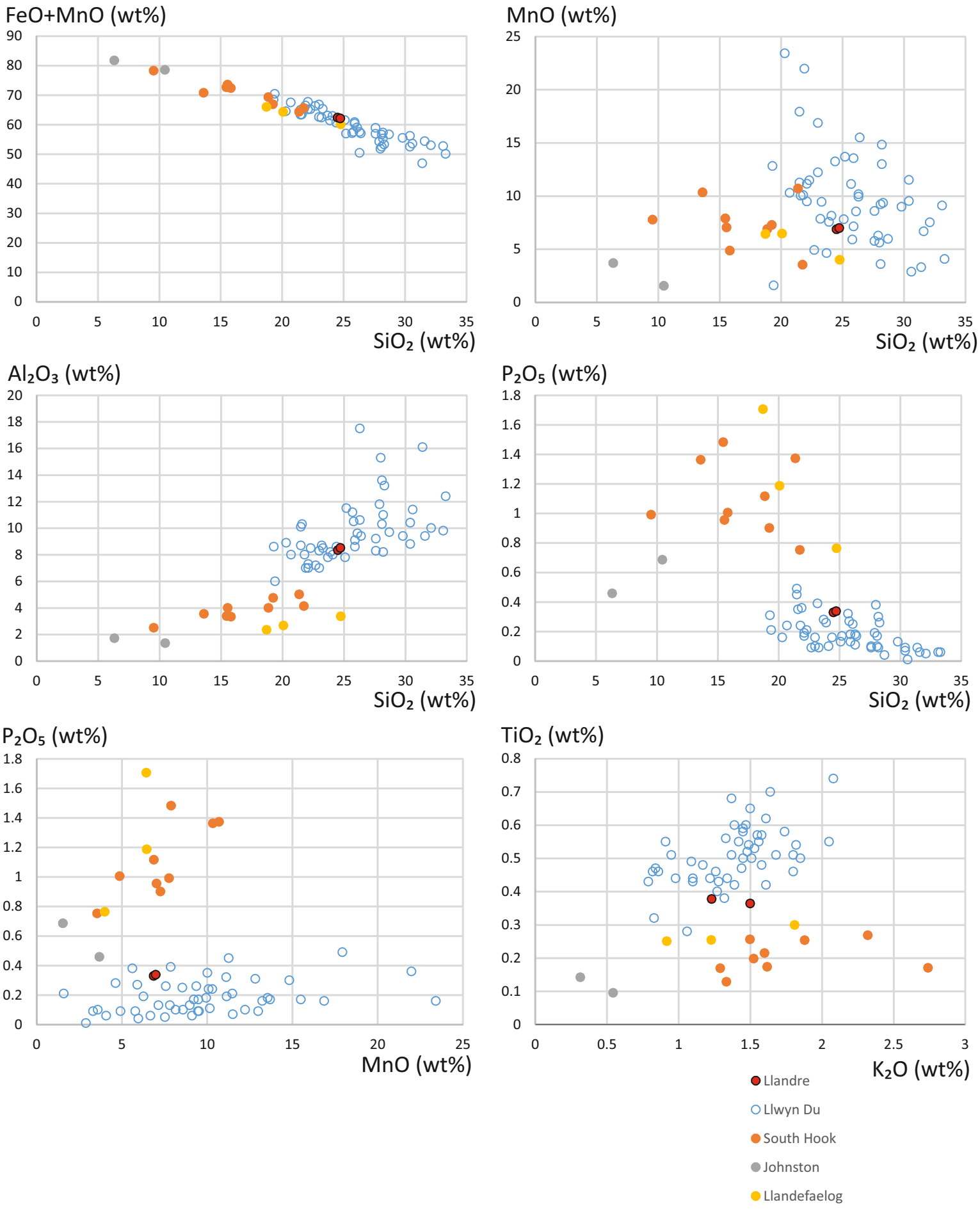
Yb 172
Lu 175
LLE1
LLE2

Figure 3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SiO2 (wt%)
MnO (wt%)
SiO2 (wt%)
Al2O3 (wt%)
SiO2 (wt%)
P2O5 (wt%)
SiO2 (wt%)
P2O5 (wt%)
MnO (wt%)
TiO2 (wt%)
K2O (wt%)
Llandre
Llwyn Du
South Hook
Johnston
Llandefaelog

Appendix:

Raw bulk chemical analyses

Table A1: Major elements by XRF. < = below detection. All elements presented as wt% oxide. Raw data, Fe expressed as Fe_2O_3 and Mn as Mn_3O_4

	Bead	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	Mn_3O_4	MgO	CaO	Na_2O	K_2O	P_2O_5	LOI	total
LLE1	LF41664	24.01	0.37	8.17	60.44	7.23	0.36	0.76	0.39	1.21	0.32	-4.57	98.99
LLE2	LF41665	24.52	0.36	8.44	60.74	7.44	0.45	0.87	0.55	1.49	0.33	-5.80	99.62

Table A2: Minor elements by XRF. < = below detection. All elements presented as wt% oxide. Raw data, Fe expressed as Fe_2O_3 and Mn as Mn_3O_4

	Bead	SO_3	V_2O_5	Cr_2O_3	SrO	ZrO_2	BaO	NiO	CuO	ZnO	PbO	HfO_2
LLE1	LF41664	0.15	0.01	<0.005	0.01	<0.003	0.13	<0.004	<0.003	0.000	0.003	<0.005
LLE2	LF41665	0.05	0.01	<0.005	0.01	<0.003	0.15	<0.004	<0.003	0.003	0.003	<0.005

Table A3 (part 1): bulk chemical analyses by ICP-MS. All elements in ppm. bdl = below detection limit

		P	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Cu	Ga	Ge
LLE1	ICP045_21	1517.67	7523.09	7.35	2514.22	44.69	30.72	63318.95	610068.92	21.20	2.94	9.81	16.96
LLE2	ICP045_22	1473.08	8204.86	5.27	2539.50	45.75	30.83	65666.46	624058.45	21.44	2.64	9.77	17.06
BCR-1	ICP045_39	1222.93	65025.72	34.17	11304.58	407.60	bdl	1590.38	126456.13	36.40	13.85	23.77	3.73
BCR-1	recommended	3646.00	-	32.41	22420.00	410.00	13.50	1838.00	-	37.55	19.60	22.19	1.40

Table A3 (part 2): bulk chemical analyses by ICP-MS. All elements in ppm. bdl = below detection limit

		Rb	Sr	Y	Zr	Nb	Mo	Ru	Sn	Cs	Ba	La	Ce	Pr	Nd	Sm
LLE1	ICP045_21	27.37	83.50	11.40	83.20	10.34	3.61	0.01	0.18	1.47	507.39	9.69	26.48	2.74	12.23	2.73
LLE2	ICP045_22	21.37	87.36	8.72	85.61	10.39	3.68	0.01	0.22	1.65	553.95	11.10	24.91	3.03	13.56	2.64
BCR-1	ICP045_39	47.44	309.93	36.82	191.34	12.33	1.82	0.00	0.70	0.98	672.40	24.00	49.70	6.40	29.40	6.91
BCR-1	recommended	46.61	334.90	-	190.30	-	1.52	0.00	-	0.96	683.30	25.46	53.94	6.77	28.68	6.60

Table A3 (part 3): bulk chemical analyses by ICP-MS. All elements in ppm. bdl = below detection limit

		Eu	Gd	Tb	Gd	Dy	Ho	Er	Tm	Yb	Lu	Hf	Ta	W	Pb	Th	U
LLE1	ICP045_21	0.62	2.51	0.35	2.11	0.44	1.28	0.18	1.41	0.18	2.09	0.41	0.58	1.62	3.16	1.06	0.62
LLE2	ICP045_22	0.54	2.35	0.33	1.98	0.40	1.14	0.16	1.14	0.15	2.14	0.38	0.50	1.40	2.94	1.09	0.54
BCR-1	ICP045_39	1.87	6.68	0.92	6.50	5.97	1.20	3.60	0.47	3.46	0.49	4.37	0.57	0.48	12.77	5.33	1.61
BCR-1	recommended	1.96	6.73	1.06	6.73	6.39	1.27	3.66	0.54	3.38	0.50	4.92	0.79	0.43	13.44	5.79	1.68

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