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Analyses of ores and residues from Dunkeswell, Devon

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Abstract

This project entails description and analysis of iron ore samples from excavations of ore-extraction pits at Dunkeswell, Devon. Some of the ore samples were obtained from in-situ deposits; others were reworked into archaeological deposits. In addition a single block of unstratified bloomery iron smelting slag (probably tapped slag) was also analysed.

The iron ores are high-grade boxstones, with laminated cavity linings of 95-96% slightly hydrated iron oxide-hydroxide (perhaps goethite). The ores are characterised by both high phosphorus (P_2O_5 contents of 0.6 to 1.1 wt%) and manganese (MnO contents of 0.3 to 1.0 wt%).The silica content of the ores is less than 2 wt%.

The trace element content of the ores is generally low. The rare earth elements (REE) shows marked depletion of the light REE compared with the Upper Crust standard. The total of the REE is low, at 27 to 59 ppm. The uranium to thorium ratio is in the range of 2 to 5.6, with very low thorium contents.

The block of smelting slag shows chemical characteristics broadly compatible with derivation from ores of the type found on the site. It is very iron-rich (at the upper limit of iron content for the medieval smelting slags from nearby Hemyock), suggesting a limited degree of interaction with the furnace wall. Concentrations of both phosphorus and manganese are also at levels elevated little, if at all, above those of the ore. The REE profile is similar to, but slightly flatter than, those of the ores, with a Σ REE of 86ppm. These features, if representative, all suggest a very low iron yield from the smelt. Tentative modelling of the likely mass balance indicates a possible yield as low as 20% of the available iron.

The project has reviewed the evidence for the ore source for the early medieval smelting at the nearby smelting centre at Hemyock. The properties of the Dunkeswell materials were compared with those from two sites in Hemyock: Culmstock Road (HCR) and Churchills Farm (HCF). The three sites are all distinct. HCF produced a single piece of ore, morphologically similar to some from Dunkeswell, and with somewhat similar chemical composition, except for elevated copper and zinc. The apparent ore samples from HCR were of different lithology to those of the other sites, probably deriving from the Upper Greensand. The chemical composition of the HCR slags, although broadly similar to those of the other sites.

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Methods

The materials were examined visually (with a low-powered hand lens or microscope when required) and given a rapid assessment. A catalogue of the examined materials from Dunkeswell is presented in Table 1.

The analytical programme was developed to address the characterisation of the iron ore, with a particular view to examining whether such ores might have supported the early medieval iron smelting industry in nearby valleys (particularly at Hemyock). Analysis of the iron smelting slag specimen was undertaken to investigate compatibility with the iron resources of the site and also for comparison with slags from the Hemyock sites. The scale of the investigation was limited to these aims, rather than a full geological investigation of the ores, and accordingly a programme of works was developed entailing bulk chemical analysis of a suite of materials ad excluding petrographic investigation.

The selected samples, four samples of ore (DUNK1-4) and one smelting slag (DUNK5) were slabbed on a diamond saw and subsamples were crushed for preparation of whole-sample chemical analyses.

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 ThermoElemental X-series Inductively-Coupled Plasma Mass Spectrometer (ICP-MS) in the School of Earth and Ocean Sciences, Cardiff University (this also generates lower quality results for Fe, Mn, Ti, P that are used mainly for QA purposes). The results of the chemical analyses are presented in full in the archive appendix (Appendix A), with the key data presented as tables in the body of the report.

This project was commissioned by Damian De Rosa of Cotswold Archaeology.

Background

The material described in this report derives from excavations in an area of intensive pitting for iron ore to the NE of Dunkeswell airfield (centred on ST137082).

The site lies on high ground (250m OD) in an area in which the 'clay with flints' overlies the Upper Greensand. The site lies 5km south of the village of Hemyock, where there have been two recent excavations of early medieval bloomery complexes which have provided evidence for ore provenance.

Previous detailed investigation of the resources exploited in the Blackdown Hills is largely restricted to excavation of ore pits at North Hill, Broadhembury (ST09900675; Reed 1997). These excavations produced two 14C dates (here recalibrated to current standards) of AD540-660 (charcoal) and AD1-230 (carbonised material in soil), from a deposit (c550) close to the base of the excavated pit.

Results

Iron ores

The ore fragments are pieces of dense boxstone crusts, probably of goethite. The laminated brown crusts are very fine-grained, commonly hard and may demonstrate a convoluted texture. The fragments provide little evidence for the original scale of the boxstone texture, but suggest structures within the range of 50-200mm, and some examples may have been larger. There is no recorded information about the completeness of the boxstone structures within the natural deposits, but the nature of the samples suggests they were fragmented pieces.

There are rare examples of crusts enclosing small white clasts up to a few millimetres across (e.g. from c132). These clasts are soft and may be highly altered lithic clasts, possibly chert. There is no other indication of the nature of the host sediment.

Internally, most boxstone fragments just enclose a pale, almost white, clay, but there are a few examples of somewhat ruddle-like iron oxide rich deposit (e.g. from c315).

Several pieces (e.g. examples from c103, c125) show zones within, or outside, the laminated goethite in which the iron oxide bears small (<0.5mm) cavities. These suggest dissolution of mineral, possibly a carbonate mineral, but perhaps more likely a sulphide.

The chemical composition of the analysed ores approaches that of pure iron III oxide-hydroxide with iron expressed as FeIII oxide plus the loss on ignition totalling 95-96wt%. The loss on ignition (LOI) was measured as 12.22-12.82 wt%, a value that is higher than the LOU of 10.13wt% that is obtained by converting an anhydrous iron oxide-hydroxide (FeO.OH) to iron II oxide (Fe_2O_3). It is likely therefore that a degree of hydration may be present – and may

be one of the factors capable of generating the slight colour banding in the ore.

The significant impurities are manganese (with a range of 0.19-0.96wt% MnO) and phosphorus (with a range equivalent to 0.6 to 1.1wt% P_2O_5). The low level of other impurities is reflected, for instance, in a low concentration of the rare earth elements (REE), with total REE of 27-59ppm. As is typical of goethite ores, the thorium content is low (0.4 to 1.1 ppm). The uranium content is higher, but still low (1.5 to 2.2 ppm).

Two of the samples (DUNK1 and 2) show mildly elevated zinc contents (569.6ppm and 142.4ppm respectively), a feature also noted in an ore fragment (sample HCF24) from the Churchills Farm site in Hemyock (Young 2015).

The upper crust-normalised REE profiles (Figure 1a; normalisation after Taylor and MacLennan 1981) show the heavy REE (HREE; Dy and above) to be slightly below parity, but horizontal, whereas there is marked depletion of the light REE (LREE; particularly Eu and below), with marked negative cerium anomaly. The profiles for samples DUNK2, 3 and 4 differ in degree of depletion but are almost parallel, that for DUNK1 is slightly flatter and has a lower degree of depletion.

Iron smelting slag

The single piece of bloomery iron smelting slag (DUNK5) was a 8720g block of very dense tap slag cake, with a neat, strongly convex base, indicating tapping into a small pit; this is probably most of the original slag cake. The cake would have had a roundedly equant shape in plan, 180mm across and 100mm deep, with 70mm of the thickness in contact with the substrate and 30mm as a slightly convex top with flow lobes of very variable width.

The chemical composition of the slag is very iron-rich, with analyses showing 80.9wt% Fe_2O_3 (suggesting approximately 79.5wt% as FeO in the original slag), with 0.84 wt% MnO and 0.87wt% P_2O_5. In general, the elemental composition shows a high degree of similarity with the ores from the site. The REE total 86ppm.

The upper crust-normalised REE profile is horizontal through the HREE (Gd and above), with depleted LREE and a negative cerium anomaly (Figure 1a). Neither the degree of depletion nor the magnitude of the cerium anomaly is as great as in the ore samples, as would be expected as a result of the mixing of the ore and furnace ceramic during smelting.

Interpretation

The nature of the ore

The analysis of the ore specimens clarifies the nature of the resource being exploited (detailing its high grade, variably elevated manganese and elevated phosphorus), but only adds a little to understanding of its geological origin.

The ores recovered during this project are fragments of boxstones – zones of iron enrichment formed when reduced iron released by weathering reactions passes into oxidised zones around joints or the outside of concretions (typically zones influenced by meteoric waters) where the iron II oxidises to insoluble iron III and precipitates. Such textures may form along the joints in a well-jointed rock, or may form around the outside of individual concretions. The fragments present in this study were too small to differentiate these possibilities with any certainty, but Reed (1997) reported on similar ores from a location 4km WSW of the Dunkeswell site and illustrated 'nodules' of up to approximately 400mm and weighing 30kg. The exteriors of the boxstone lacked any adhering rock that might have given a clue as to the context of their formation.

The relatively 'clean' interiors of the boxstones,' would also support a derivation from concretions with a high proportion of cement that has dissolved during weathering. The high iron content of the crusts may indicate that the concretions were originally of siderite (or perhaps ankerite or strongly ferroan calcite). The evidence for zones of cavities suggesting possible minor sulphide mineralisation towards the outside of the concretions may be significant in determining the environment of generation of the concretions.

The origin of the Clay with Flints has been controversial. Gallois (2009) has provided a comprehensive review, demonstrating that large scale dissolution of the Chalk (and locally the calcareous part of the Upper Greensand) has been the major mechanism, which together with subsequent deposition of further sediments during the Late Paleocene to early Eocene produced the Clay with Flints *sensu stricto*. Later, further weathering and deposition during the Pleistocene generated the Clay with Flints *sensu lato*.

Isaac (1981) has produced a detailed study of the Tertiary sedimentary component of the Clay with Flints, and has documented a variety of deposits and environments, including palaeosols and silcretes.

There would appear to be two potential origins for the boxstone ores of Dunkeswell: (1) they were formed from concretionary rocks originally present within the Upper Greensand and left as *remanié* clasts with the Clay with Flints, or (2) they formed diagenetically within the Tertiary component of the Clay with Flints.

Distinguishing with certainty between these two scenarios is not possible on the present evidence. Iron-rich carbonate concretions have not been recorded elsewhere within the Clay with Flints' Tertiary deposits, nor have they been described from the Upper Greensand. Clearly, more geological research is required to establish the precise origin of this significant resource.

The slag from Dunkeswell

There is no dating evidence for the tapslag block (DUNK5), although its equant shape is more reminiscent of some Roman period slags, rather than the more elongate flows usually formed within the tapping channels of typical medieval slag-tapping furnaces.

The shape of the REE profile is intermediate between those of the ore samples and a horizontal profile that would be characteristic of a furnace ceramic.

Modelling a smelting mixture of an ore of composition equivalent to DUNK4 (the richest of the ore samples) with an idealised ceramic of upper crust composition has a best fit for the REE at 74% ore and 26% ceramic with an enrichment of 1.23. Such a clay-rich mix is plausible (particularly if the ore was not as clean as the analysed samples), but such an enrichment factor is

equivalent to the production of no iron metal – merely the dehydration/dehydroxylation of the goethite and reduction to an iron II oxide.

The concentrations of both phosphorus and manganese are also at levels close to those of the ore samples, again suggesting a low iron yield.

If the sample was representative of the smelt as a whole, then it would therefore seem likely that either it was extremely unproductive smelt, or was not produced from ore of precisely the sampled composition, although the exploited resource must have been of very similar type. It is possible, however that the slag sample taken from the margin of the cake was not representative of the bulk composition of the slag from the smelt, but may have been produced early in the reaction when little iron metal was yet being produced.

The resources for smelting at Hemyock

The two recently-investigated sites in Hemyock, Churchills Farm (Young 2015) and Culmstock Road (Young 2014; Rainbird and Young in prep.) each produced internally coherent suites of slags, with some associated ore. The REE profiles are illustrated in Figure 1b (Culmstock Road) and 1c (Churchills Farm). In each case the grey tone on the figure shows the range of composition of the slags and the ore samples are shown as black lines.

For the later 9th-11th century site at Churchills Farm, the single ore fragment (sample HCF24) recovered gave a REE profile with a shape very close to those of the analysed slags and with an overall REE content at the lower limit of those of the slags. The profiles show a broadly horizontal section across the HREE (with a slight central dip), and depleted LREE and a strong negative cerium anomaly. Such a profile shows some strong similarities with those of the samples from Dunkeswell.

One significant difference with the ores from Dunkeswell, was that analyses of all the materials from Churchills Farm, ore and slags, showed elevated levels of copper (330-480ppm, compared with 18-80ppm for slags from Culmstock Road and only 6ppm for the slag from Dunkeswell). The most likely explanation for raised copper would be the influence of sulphide minerals on the ore composition). Some caution was applied to this observation, for although close to the lower limit of detection analysis of the Churchills Farm samples by WD-XRF failed to show the same enrichment in copper seen in the more sensitive ICP-MS analyses.

The sample of ore (HCF24; see Table 2) was a brown-yellow boxstone with a pale clay fill overlying a thin ruddle layer – all properties of the Dunkeswell ores. There is thus strong evidence that Churchills Farm was exploiting a resource of the same type as that at Dunkeswell (i.e. boxstones from the Clay with Flints).

In contrast, the earlier of the Hemyock sites, the 7th-9th century site in Culmstock Road (Young 2014) produced nine pieces of low-grade ore (Table 2), six of which were pelletal, two fossiliferous and one a micaceous siltstone. Analyses reported on in full by Young (2014) and Rainbird and Young (in prep) were taken from one pelletal piece (sample HCR8) and one apparently roasted pelletal rock (Sample HCR9). The match between the REE profiles of the ores and the slags was less good than for the other material discussed here. The slag profiles show relatively depleted LREE, but a tendency for progressive relative enrichment in the HREE, rather than a plateau. The roasted sample shows strongly elevated REE with a humped profile centred on gadolinium – quite unlike that of other materials discussed here and incompatible with being representative of the bulk composition of the ores employed during the smelting that produced the slags. The raw pelletal rock (HCR8) is a better, but still imperfect match for the slag profiles, having a flatter profile than the slags, but similarly inclined.

It is therefore clear that the site at Culmstock Road smelted ore from a different resource than did the other sites. The ore samples from the site were pelletal and apparently from the glauconitic parts of the Upper Greensand. It is possible that the samples were from iron-rich weathering crusts, rather than the bulk, raw, Upper Greensand glauconitic rocks, that would probably be of rather low grade. A situation in which rocks of Upper Greensand origin occurred in a derived context within the Clay with Flints is plausible, but there is no evidence that this was the case.

In summary, the Dunkeswell site demonstrates the high grade of the iron ores present in the Clay with Flints in this part of the Blackdown Hills. As well as the quarrying of ore, some smelting of the ore had taken place in the area. The site at Churchills Farm was exploiting a similar ore, but with an apparently elevated copper content – possibly suggesting an increased role of sulphides in the concretions. In contrast, the site at Culmstock Road had smelted an entirely different ore from the Upper Greensand.

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Caption

Figure 1: Upper Crust normalised rare earth element profiles (normalisation after Taylor and MacLennan 1981).

(a) samples from Dunkeswell. Ores are shown as black lines, the slag as a pink line.

(b) samples from Culmstock Road, Hemyock. The field of analysed slag compositions is shown in grey tone, analyses of ores as lines.

(c) samples from Churchills Farm, Hemyock. The field of analysed slag compositions is shown in grey, an analysis of an ore as a black line.

Context	Feature	Sample wt (g)	Item wt (g)	Item number	Notes	Sampled as:
u/s			8720		part of a very dense tap slag cake, with neat strongly convex base, indicating tapping into a small pit; this is probably most of the tapped material	DUNK5
103		1255	1175	10	ferruginous boxstone	
			70	1	chert	
			10	1	broken pebble with reddish porous ferruginous, slightly granular material - similar to possible sphaerosiderite material from 125	
109		945	895	13	ferruginous boxstone	
			50	3	chert	
125		3235	3040	21	ferruginous boxstones	DUNK2
			70	5	cherty pale rocks	
			125	4	dense ferruginous rocks, possibly pelletal (one then broken to inspect interior - dense goethite with strings of tiny cavities, near margin shows dark blebs with pale matrix - this might just be oxidised sphaerosiderite or pyrite?)	DUNK4
127		2930	2440	19	ferruginous boxstone	
				8	flint	
129		245	160	3	ferruginous boxstone	
			85	2	flint	
132		220	220	2	boxstone fragments, with one with clastic outer layers apparently including chert fragments	
133		1920	1920	4	rich boxstone fragments. In one the inner botryoidal layer is so black blue it may suggest considerable manganese	
135		1260	1255	3	ferruginous boxstone	
135		1465	1465	3	complex ferruginous boxstones	
302	natural	725	725	1	excellent example of boxstone with convoluted inner coatings	DUNK1
304	ditch 303		960	4	complex ferruginous boxstones	
312	pit 311		475	1	ferruginous boxstone	
315	pit 314	975	100	1	fragment of flinty-textured boxstone with ruddle like botryoidal lining	
			870	2	ferruginous rocks, not certainly true boxstones but involving ferruginous crust-like zones	DUNK3

Table 1: Catalogue of supplied materials from Dunkeswell

sample	context	feature	wt (g)		notes
HCR8			255	after cutting	coarse pelletal rock with pale matrix and large yellow-brown oxide peloids. Ferruginous charcoal/wood rich concretion on one end
HCR9			488	after cutting	red pelletal ore, with oxide lined rounded cavities. Interpreted as roasted weathered greensand, resembles some Tisbury material
HCR unused #1			1845	after cutting	porous yellow brown rock with pale yellow-brown void fills and dark brown crusts - some of the crusts are curved and serrated and may be fossils. This looks like an oxidised and weathered concretion - possibly originally sulphide.
HCR unused #2			275	after cutting	Finely-laminated micaceous ferruginous mudrock with superficial manganese oxides. One margin is a cross cutting crinkled zone, possibly a minor fault or cavity margin
HCR colln of ore			195	as found	coarse pale pelletal rock with pale matrix and yellow-brown iron oxides. The pellets are very soft. Probably a weathered greensand, but could be a pedogenic material.
			180	as found	fine pelletal rock, variably weathered but locally dark green in small areas amongst the dominant yellow brown, some sand. Weathered greensand.
			120	as found	fine, massive, yellow-brown mudrock with ?bioturbation. One broken surface appears to show contact with pelletal lithology
			45	as found	yellow brown, massive yellow brown lithology, with some slightly darker brown crusts around cavities
			10	as found	broken fragment of complex boxstone texture with multiple cavities, dark brown iron rich rims. Possible conglomeratic texture with white inclusion (probably a fossil) on one side. Some yellower patches show a possible weathered pelletal structure on one surface
HCF24	790	Furn 789	70	after cutting	Boxstone with a dark crust with a slight yellowish core; the, interior has a light red ruddle layer a millimetre thick and a void fill of pale clay.

Table 2: Ore samples from sites in Culmstock Road and Churchills Far, Hemyock (see also Rainbird and Young in prep, Young 2014 and Young forthcoming)

Table 3: Major elements by XRF. The shaded column shows the effect of expressing the iron oxide as FeO. The loss on ignition has been included in the total where positive. A negative loss on ignition (i.e. a gain in weight on ignition) has not been included in the expressed total. < indicates the element was below detection.

		SiO_2	Al_2O_3	Fe_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	LOI	total
DUNK1	good boxstone	1.85	1.13	82.86	74.57	0.58	<0.007	<0.003	<0.010	0.04	0.07	1.096	12.22	*100.02*
DUNK2	boxstone	1.77	0.92	83.24	74.92	0.29	<0.007	<0.003	<0.010	0.03	0.06	0.555	12.73	*99.70*
DUNK3	ferruginous rock with crusts	1.53	1.03	84.03	75.63	0.19	<0.007	<0.003	<0.010	0.04	0.06	0.694	12.47	*100.14*
DUNK4	holes	0.75	1.33	82.74	74.47	0.96	<0.007	<0.003	<0.010	0.00	0.06	1.106	12.87	*99.97*
DUNK5	tapped slag	10.70	3.64	80.97	72.87	0.84	0.15	0.49	0.01	0.36	0.24	0.866	-6.58	*98.47*

Table 4: Major elements by ICP-MS. All values as wt% oxides.

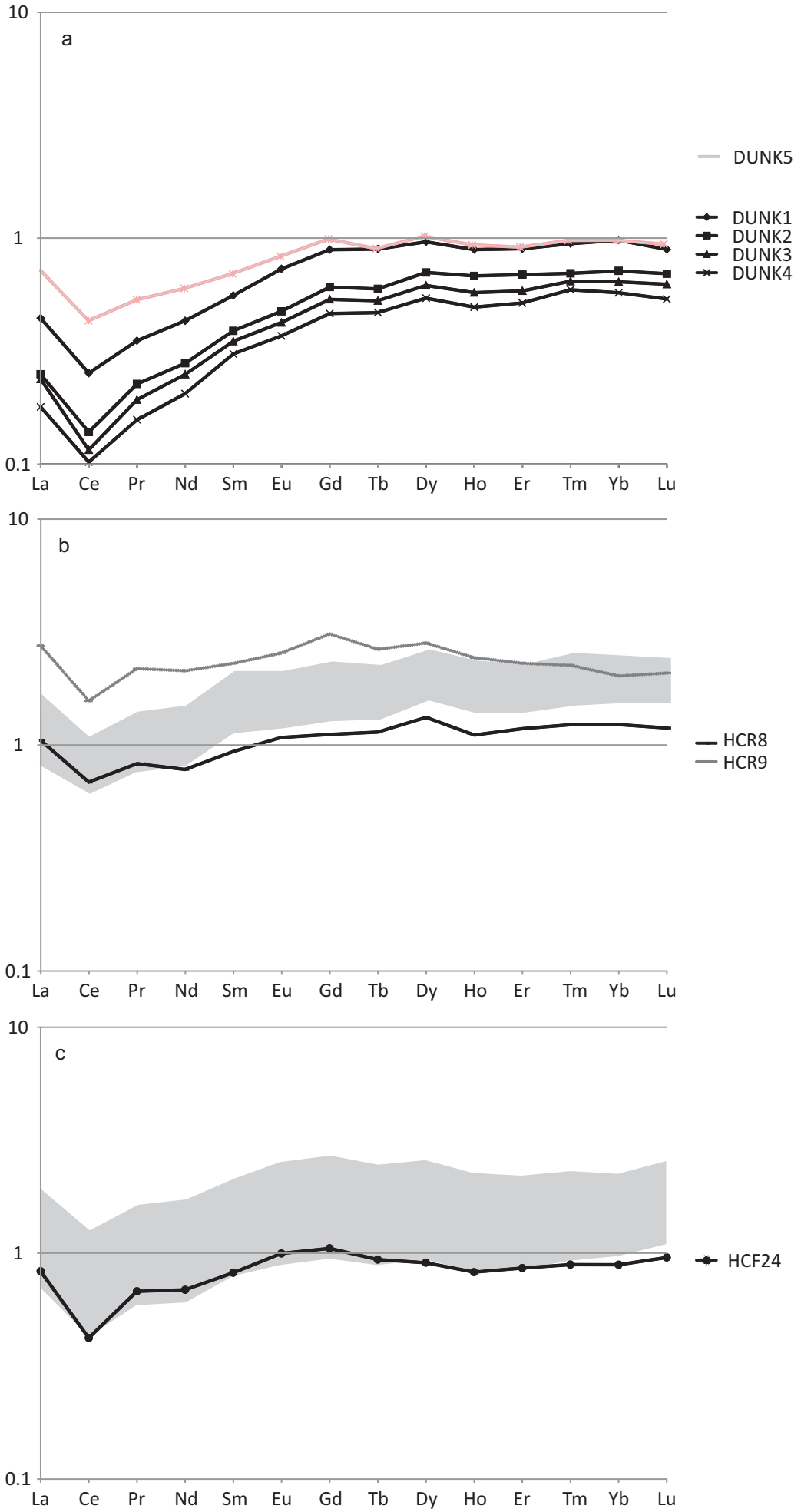
		SiO_2	MnO	FeO	TiO_2	P_2O_5
DUNK1	good boxstone	2.17	1.56	90.11	0.04	1.12
DUNK2	boxstone	2.06	1.57	90.73	0.05	0.54
DUNK3	ferruginous rock with crusts	5.73	1.62	90.78	0.05	0.66
DUNK4	holes	6.99	1.67	87.37	0.04	1.13
DUNK5	tapped slag	1.82	1.55	89.75	0.25	0.84

Table 5: Trace and minor elements by ICP-MS. All values in ppm.

	Sc	V	Cr	Co	Ni	Cu	Zn	Ga	Rb	Sr	Y	Zr	Nb	Mo	Sn	Cs	Ba
DUNK1	10.0	53.5	21.4	46.1	55.7	26.2	569.6	2.7	4.4	15.8	34.5	74.8	1.78	0.36	3.26	0.60	61.70
DUNK2	6.4	34.1	14.5	110.1	15.6	24.1	142.4	2.2	3.9	15.6	25.4	71.8	1.68	0.39	2.93	1.35	53.87
DUNK3	13.2	31.2	11.1	24.1	7.2	8.7	69.2	1.1	2.0	18.4	47.7	71.5	0.32	0.14	0.72	0.43	27.82
DUNK4	14.0	39.5	9.4	27.6	17.2	3.4	56.2	1.2	0.7	14.1	39.4	70.1	0.19	0.29	0.60	0.08	28.37
DUNK5	22.3	60.0	39.7	22.8	b.d.	6.5	10.2	2.6	6.6	72.2	73.9	148.4	2.32	0.15	0.44	0.54	67.75

	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Hf	Ta	Pb	Th	U
DUNK1	13.24	16.14	2.49	11.17	2.50	0.64	3.36	0.57	3.36	0.71	2.06	0.31	2.15	0.28	1.87	0.27	6.58	1.11	2.20
DUNK2	7.47	8.86	1.60	7.25	1.74	0.42	2.30	0.38	2.46	0.54	1.58	0.23	1.57	0.22	1.69	1.10	5.54	0.82	2.02
DUNK3	7.12	7.39	1.37	6.48	1.57	0.37	2.03	0.34	2.15	0.46	1.34	0.21	1.41	0.20	0.82	0.06	2.88	0.58	1.50
DUNK4	5.36	6.53	1.11	5.32	1.38	0.32	1.76	0.30	1.89	0.39	1.18	0.19	1.26	0.17	0.85	0.00	2.43	0.39	2.21
DUNK5	21.52	27.55	3.78	15.54	3.12	0.73	3.75	0.57	3.56	0.74	2.09	0.32	2.15	0.30	1.90	0.24	1.38	1.44	3.15

Figure 1


a
10
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La Ce Pr Nd Sm Eu Gd Tb Dy Ho Er Tm Yb Lu
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