Assessing isotopes as a means for the provenancing of ancient iron artefacts (NEIF application – Steve Harding, Mark Pearce, Chas Jones & Jane Evans, 10/21)

1. Introduction. Lead isotope analysis (LIA) has shown itself to be the most effective tool for the provenancing of ancient metal artefacts of silver and copper and its alloys[1] but its usefulness for the provenancing of iron artefacts has not been established. Recent research has proposed the use of LIA together with trace element patterns of slag inclusions[2], LIA in combination with Sr isotopes[3], or an alternative combination of Os and Sr isotopes[4]. Moreover, it has recently been proposed that Fe isotopes may be useful for provenancing iron artefacts[5]. The approach taken by previous studies has been to concentrate on analysing material from a single site and the ores which are thought likely to be the source of the iron[2, 3, 4] rather than comparing artefacts from a selection of sites. Iron ores are extremely widespread throughout the British Isles[6] and consequently in many cases it is difficult to identify the likely sources of the iron ore used at specific sites, especially in later periods when iron circulated more widely. After the collapse of Roman Britain, it is thought that iron was initially obtained from bog iron during the Saxon period, with the mining of iron ore restarting in the later part of the period[7].

2. Research Hypothesis and Aims. Our hypothesis is that it is possible to use isotopes to characterize ancient iron artefacts from the Viking Age. Our primary aim is to test this hypothesis by studying objects from a selection of archaeological sites using isotopes of Pb, Sr and Fe, in association with trace element patterns (such as phosphorus and manganese). Pb and Sr isotope analyses will be done through the NEIF scheme with Dr. Jane Evans, while Fe isotope analyses will be carried out in parallel in France by Dr Jean Milot, who has pioneered the use of Fe isotopes in provenancing[5]. Artefact geochemical compositions will be determined by ICP - QMS as part (and funded separately by) a PhD at the University of Nottingham. Fe isotope work will be funded by internal University of Nottingham and French funds. Because of the ubiquity of iron ores, we do not believe that their study can be undertaken at this stage of the project, but we shall include samples of bog iron from the important and well-documented[8] source in Foulness Valley (East Yorkshire), which is known to have been exploited since the later first millennium BC. This will also enable us to address our second aim which is to provide the platform for a further study to establish whether bog iron has a specific isotopic signature which can be compared to the iron ores for which published data is available. Our final aim is to help stimulate the growth of databases for artefact provenancing in conjunction with an RSC Meeting.

3. Background, current research and challenges. We have already undertaken a pilot study in collaboration with Dr Jean Milot (Toulouse/Lyon) & Professor Jane Evans (BGS) using University funds in order to evaluate the project feasibility (see Appendices). Iron artefacts are often highly corroded, but we have shown that it is possible to determine their Pb, Sr and Fe isotopes from clean material drilled from below the corrosion (Appendix Fig.2). The pilot study examined the Pb, Sr and Fe isotopes of 10 Viking Age artefacts from Fulford in N. Yorks (possible location of the AD 1066 battle), Bebington Heath on Wirral (possible location of the AD 937 Battle of Brunanburh) and Meols (a Viking seaport) (Appendix Fig. 1). Artefact compositions were determined by ICP-QMS at the University of Nottingham (Appendix Fig.3). Our pilot study showed that definite patterns exist in the data, Meols seems to separate in Sr vs Ba and Cu vs Zn, while Wirral/Bebington Heath seems to separate in Co vs Ni, but the number of samples was much too low to understand their significance, given the lack of isotope studies on iron artefacts. The high range of $^{87}Sr/^{86}Sr$ values, beyond what would be expected for bog iron (with a cut-off around 7.09), suggests that mined ore was being used, a preliminary conclusion supported by the Fe isotope data.

4. Materials, permissions and sampling strategy. CoIs on the project are Chas Jones (Fulford Battlefield Society), Professor Mark Pearce (Classics & Archaeology, Uni Nottingham). Non-BGS project collaborators are Dr Jean Milot (ENS de Lyon, Uni Toulouse) and Dr Liz Bailey (Biosciences, Uni Nottingham). Samples will be obtained from iron artefacts from 3 early medieval sites in England: the Saxon-Viking battlefield site at Fulford in North Yorks (30 samples), the Viking

Age site at Bebington Heath/Storeton on the Wirral, Merseyside, hypothesised to be connected to the lost battlefield of Brunanburh (30 artefacts), and the Viking encampment of Torksey, Lincs, in the Trent valley (30 artefacts), together with 10 samples of bog iron in slag from the Foulness valley, East Yorks. [All permissions have been obtained and all samples are available].

Fulford (North Yorks), was the location for a battle in AD 1066 between Norse invaders and the Anglo-Saxons, immediately before the better known battle of Stamford Bridge. The archaeological material consists of iron weapons found at a number of short-lived iron recycling sites that were abandoned by the Norse victors at Fulford when they were defeated at Stamford Bridge five days later[9]. The iron material from **Bebington Heath** (Wirral, Merseyside) was recovered from the possible location of the AD 937 battle of Brunanburh between Norse-Scottish and Anglo-Saxon armies[10]. The material has been typologically assigned to the late Saxon/Viking period and shows parallels with the artefacts from Fulford. **Torksey**, Lincs, in the lower Trent Valley, was the site of the winter encampment of the Viking Great Army in AD 872-873 and iron working is documented at the site[11]. It is close to both outcrops of both bog iron and carbonate ores of the Jurassic scarp. Because of the likelihood that the Viking armies documented at Fulford, Brunanburh and Torksey used iron artefacts made of bog iron, we propose to investigate a well-known source close to Fulford and Torksey. Iron was in fact extracted from bog iron in the **Foulness** river valley, East Yorks from the later first millennium BC[8]. Samples of iron in slag produced during the smelting of bog iron have been included in the study in order to exemplify the isotope signal of bog iron. The applicants will undertake a parallel project, funded by Norwegian research funds and in collaboration with the University of Oslo, looking at the Pb, Sr and Fe isotopic signals of Viking age iron artefacts from Scandinavia. This will provide comparative data for the English material and will enhance the conclusions of this study.

***5.* Expected outcomes** 1. Analytical confirmation of the usefulness of isotope analysis for the characterisation of archaeological iron artefacts. 2. It will be the first to compare Pb, Sr and Fe isotopes, plus trace element determination, in a unified study. 3. At a minimum we will be able to group and compare data from the different sites and constrain the possible origins using the published Pb & Sr isotope data & maps. 4. Significant inroads into studying iron provenance, a hitherto unsolved problem. 5. A methodological paper on the results of the analyses (target: *Journal of Archaeological Science*, JIF 3.216). 6. Proof of concept for the preparation of a major UKRI funding bid to establish a protocol for the provenancing of iron artefacts using isotopes (likely target: NERC or AHRC). 7. Strongly fueling the population of publicly available isotope databases, the aim of our Royal Society of Chemistry meeting at Burlington House (June 2022: https://www.rsc.org/events/detail/44249/advances-in-isotope-ratio-and-related-analyses-for-mapping-migrations-from-prehistory-to-the-viking-age) at which we should like to present the preliminary results of this study.

References 1. Artioli G et al (2020). LIA of prehistoric metals in the central Mediterranean area: a review. *Archaeometry* **62**, suppl. 1: 53-85. 2. Schwab R et al (2006). The provenance of iron artefacts from Manching: a multi-technique approach. *Archaeometry* **48**: 433-452. 3. Degryse P, et al (2007). Tracing the resources of iron working at ancient Sagalassos (south-west Turkey): a combined lead and strontium isotope study on iron artefacts and ores. *Archaeometry* **49**: 75-86. 4. Brauns M et al (2010). Provenance of Iron Age iron in southern Germany: a new approach. *J Arch Sci* **40:** 841-849. 5. Milot J et al (2016). Iron isotopes as a potential tool for ancient iron metals tracing. *J Arch Sci* **76**: 9-20. 6. Tylecote RF (1962). *Metallurgy in Archaeology*: 175-179. London: Edward Arnold. 7. Hall NS (2017). *An investigation into phosphoric iron production in Eastern England*. PhD thesis, Univ. Nottingham. http://eprints.nottingham.ac.uk/id/eprint/42308. 8. Halkon P (1999). *Valley of the First Iron Masters: Landscape Archaeology in the Foulness Valley, East Yorkshire*. Hull. 9. Jones C 2012 The role of storeroom collections in the identification of battle debris. *Arms & Armour* **9**: 54-62. 10 Harding SE (2011) Locations & Legends in (Livingston M ed.) *The Battle of Brunanburh*: Univ Exeter Press pp351-361. 11. Hadley D & Richards J (2016). The winter camp of the Viking Great Army, AD 872–3, Torksey, Lincolnshire. *Antiquaries J* **96**: 23-67.

__Appendix 1. Viking Age iron objects included in the pilot study.__

10 samples were analysed in the pilot study: **Fulford, Yorkshire (4 samples)***:* Ful16-176: Repaired sword examined with CT scan found buried in recycling area; Ful03-6: Complete conserved tanged arrow from the area of King Harald's landing place; Ful16-56: Planishing anvil with mineralised wood revealed using CT scan from the ford; Ful03: Axe billet found in recycling area with Norse-style trade billet. **Bebington Heath, Wirral (3 samples)** all from the putative Anglo-Saxon camp: WA182: Triangular pommel shape; WA1074: Sword tip shape; WA700: Thin small blade. **Meols (3 samples):** ME02: Axe head; ME03: Weapon tip; ME04: Spear point shape (see: http://www.nottingham.ac.uk/-sczsteve/BBC_OurCoast_BoatClip.mp4) believed to have been excavated from a grave in the late 19th century.

Fig. 1. Artefacts sampled

Full16-156

Full03-6

Full16-56

Full03

WA182

WA1074

WA700

ME02

ME03

ME04

Appendix 2. Results of the pilot study

Fig. 2. Pilot isotope analyses. *4 iron objects from Fulford (orange), 3 samples from Wirral/Bebington Heath (green) and 3 samples from Meols (purple)*

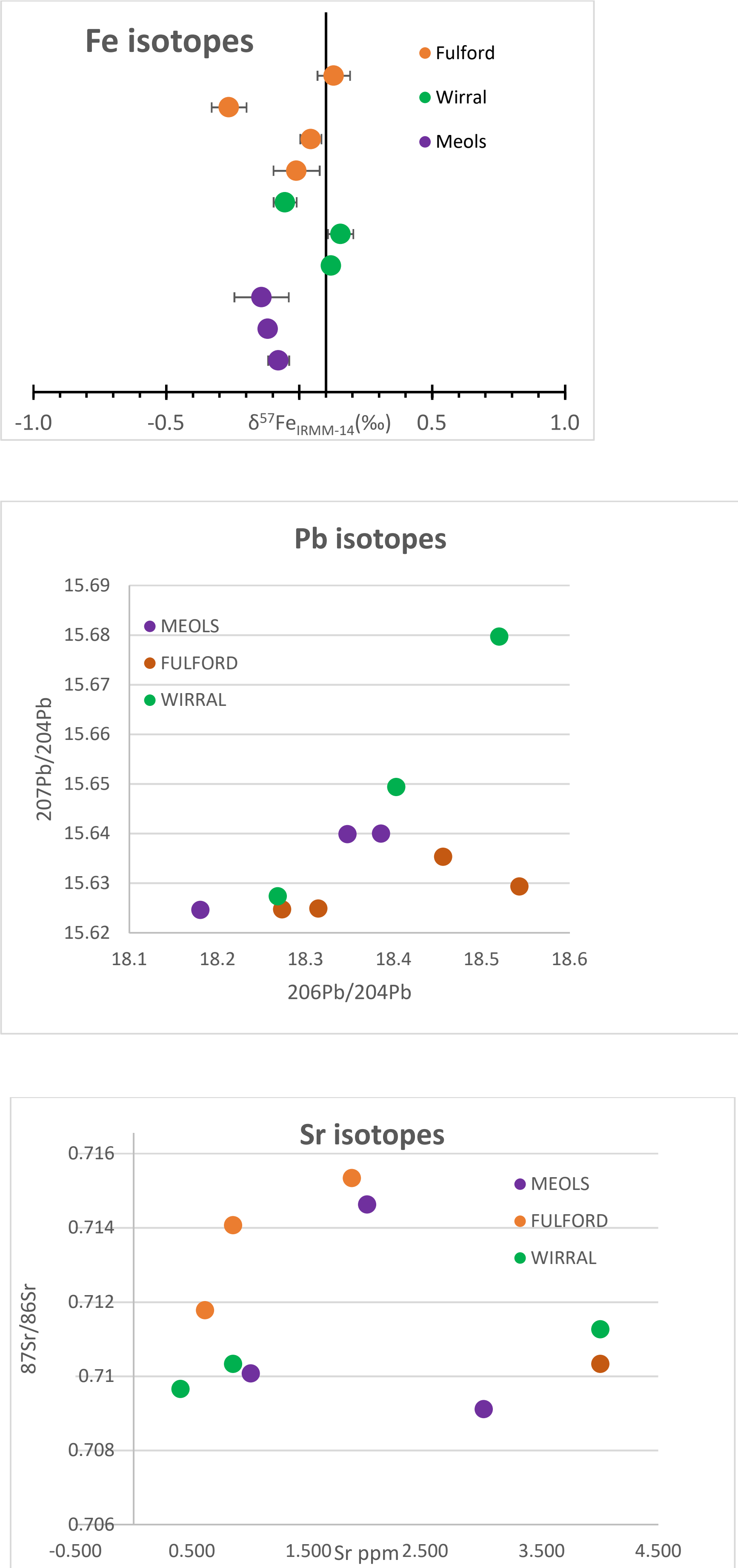
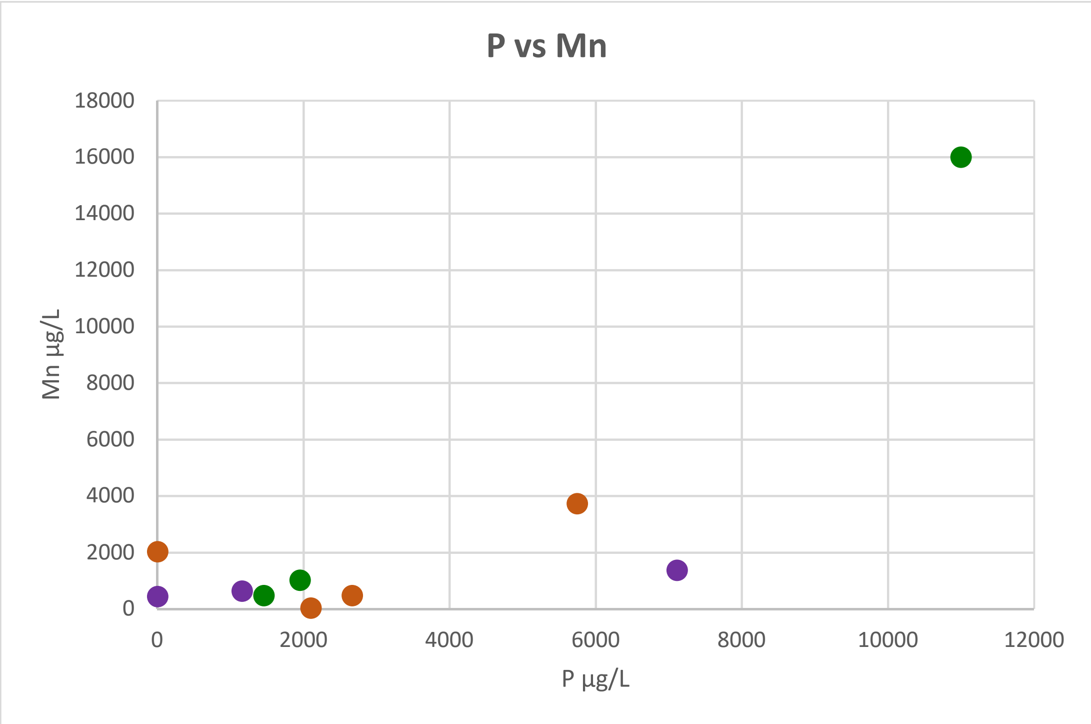
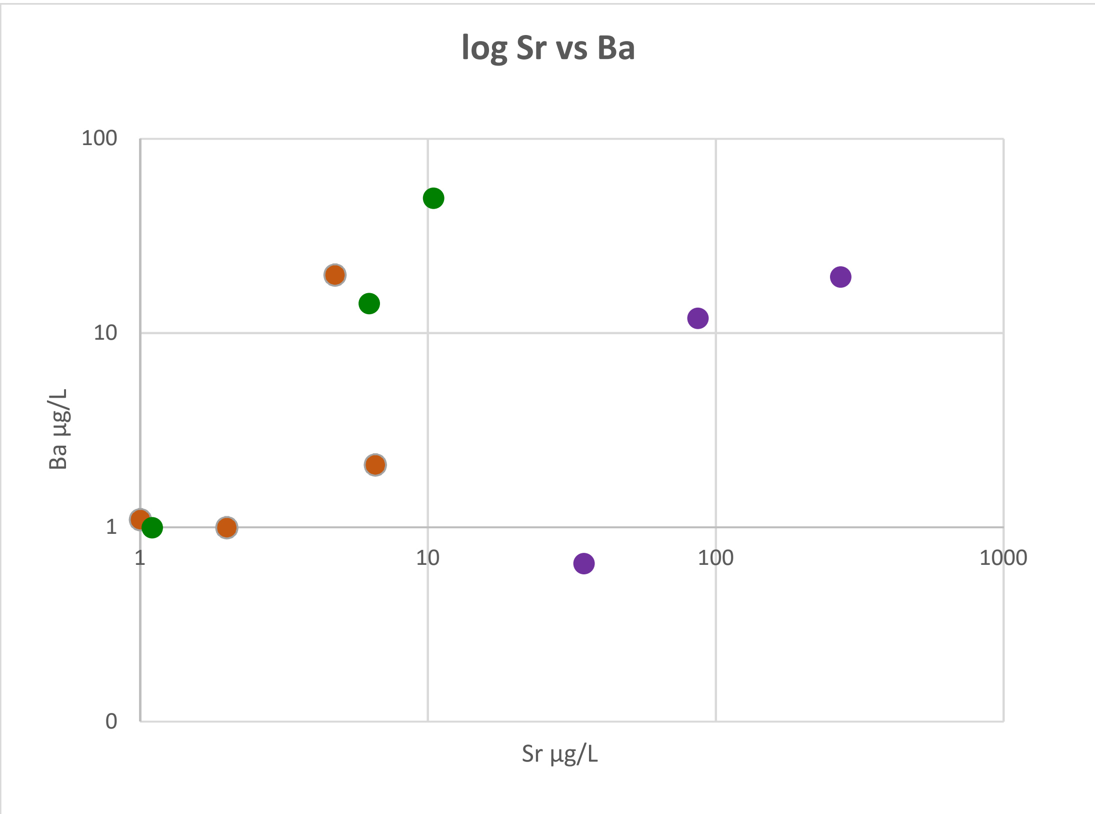
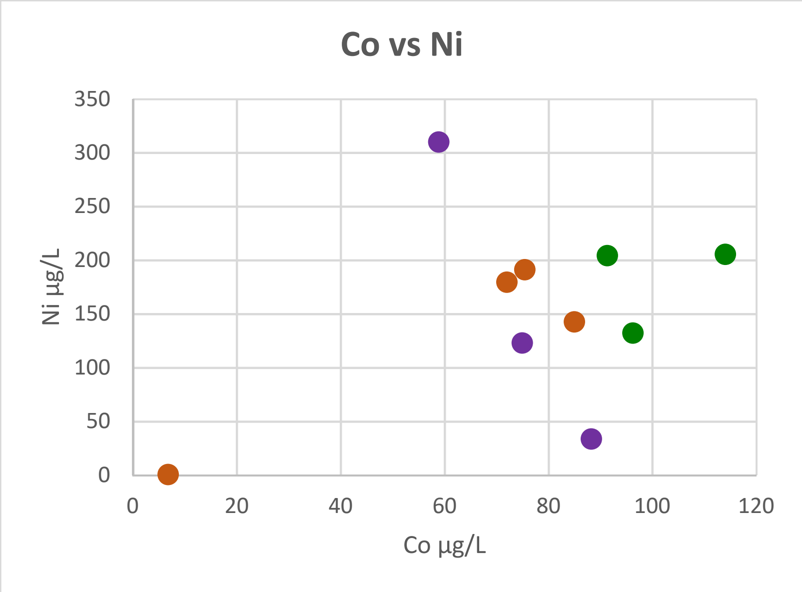


Fig 3. Key elemental composition plots (ICP-QMS analysis)

Bivariate plots for elements highlighted in previous literature[2] as likely to be informative regarding iron artefact provenance. Expressed as a % of total elemental composition (excluding sulphur). Orange Fulford, green Wirral/Bebington Heath and purple Meols. Logarithmic plots used where required by major differences in composition. Some patterns emerge (e.g. Meols seems to separate in Sr vs Ba and Cu vs Zn, while Wirral/Bebington Heath seems to separate in Co vs Ni) but a larger data set is required.






Co vs Ni
Ni µg/L
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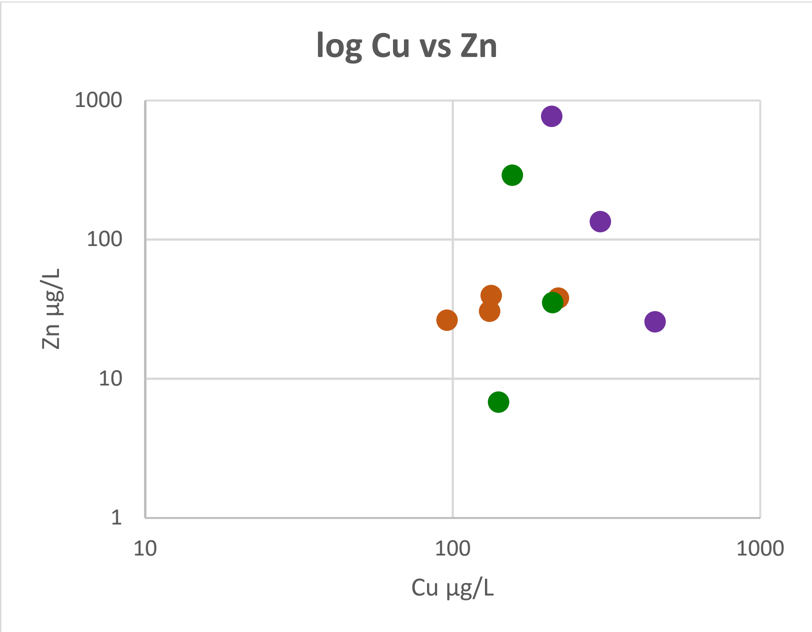
300
250
200
150
100
50
0
0
20
40
60
80
100
120
Co µg/L


log Cu vs Zn
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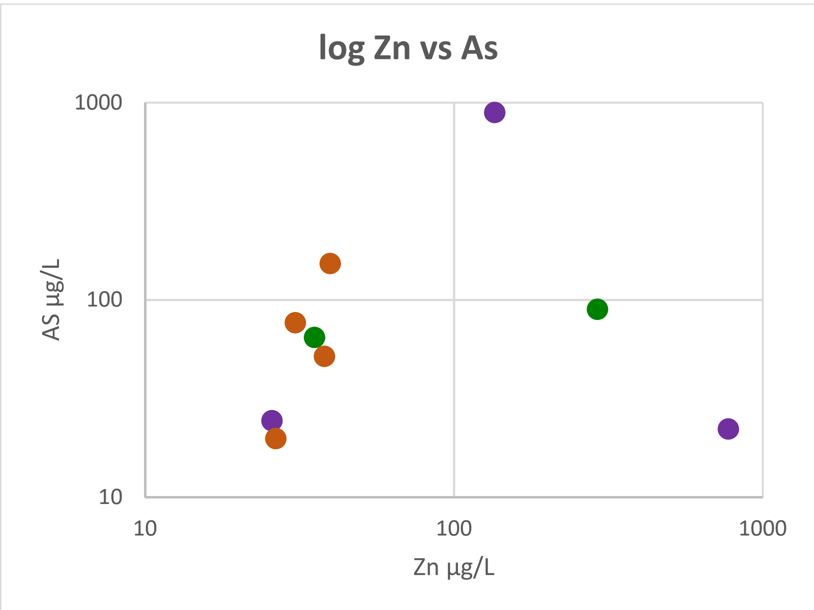
1000
100
10
1
10
100
1000
Cu µg/L


log Zn vs As
AS µg/L
1000
100
10
10
100
1000
Zn µg/L