

LIA OF PREHISTORIC METALS IN THE CENTRAL MEDITERRANEAN AREA: A REVIEW*

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Following animated discussions in the 1990–2010 period concerning the validity and potential application of Pb isotope data to yield information on ancient metallurgy, recently lead isotope analysis has been extensively applied with alternate success and difficulty to the early stages of copper/lead/silver/tin metal flow in the Central Mediterranean area, arbitrarily defined as including Italy, the Mediterranean Islands, and the surrounding regions for comparison purposes. A wealth of data are now available in the literature, many of them interpreted within local contexts and limited geographical extension, and often within a shifting conceptual modeling frame. A brief review of the recently published data indicate that the metal flow in prehistory and protohistory is far more dynamic than presumed on the basis of the traditionally assumed archaeological models. It is suggested that the isotopic tracers, if correctly applied and interpreted, may substantially help in decoding the metal exploitation and trade patterns at different scales, from the local links between mines and smelting sites to wider regional or long-distance trades. The abundant dataset available are however in need of thorough interpretation in terms of wider archaeological and archaeo-metallurgical questions, possibly by the use of advanced statistical methods and unconventional data mining protocols.

KEYWORDS: PB ISOTOPES, CENTRAL MEDITERRANEAN, METALLURGY, COPPER ALLOYS, LEAD, SILVER, TIN

INTRODUCTION

Lead isotope (LI) analysis (LIA) is conceptually rooted on the radiogenic isotopes techniques commonly used in geology and petrology to decode the age of formation of rocks, including the age of the Earth and of meteorites, providing clues on the age and evolution of the entire Solar System (Faure and Mensing 2005; Allègre 2008). Rocks and ore deposits that were generated following the common lead hypothesis (Sinha and Tilton 1973) can be chronologically discriminated based on the position of their measured Pb isotope ratios on the well-modeled universal decay curve of radiogenic elements (Cumming and Richards 1975; Stacey and Kramers 1975; Albarède *et al.* 2012). This concept was first brilliantly proposed as a method to distinguish the geological origin of archaeological metals in the mid-sixties (Brill and Wampler 1965,

*Received 20 October 2019; accepted 3 February 2020

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1967; Grögler *et al.* 1966). After more than a century of chemical analyses of ancient metals (see the extensive historical accounts in: Rehren and Pernicka 2008; Pernicka 2014; including the projects of systematic chemical analyses of ancient metals carried out in Halle and Stuttgart), the transfer of the concepts of isotope geology to archaeometallurgy represented a substantial advancement in the field of metal provenancing.

LIA were first systematically carried out on silver ores and objects, mostly from the Eastern Mediterranean and the Aegean area by the joint effort of the Oxford and Heidelberg researchers (Chamberlain and Gale 1980; Gale *et al.* 1980; Wagner *et al.* 1980). The subsequent application of the method to copper and copper-based alloys (Gale and Stos-Gale 1982), and especially the skilled combination of lead isotope ratios and trace element patterns, opened the way to the possibility of reliably relating metal artefacts to the specific ore deposits from which they could have originated, something that had been quite elusive with chemical methods alone. The trace element pattern measured on the metal does preserve some chemical information of the ores of which it was made (Pernicka 1999). However, as a matter of fact, chemical analyses by themselves mostly fail to provenance metals due to large chemical heterogeneities in ore deposits and variable fractionation of chemical elements among ores, slag, and metal in reducing procedures performed with different smelting processes and conditions. Metal refining, alloying, remelting and mixing may also complicate interpretation and often make reliable provenancing impossible by trace element analysis alone. There is ample discussion in the literature about this issue, and it will not be continued here, so the reader is invited to peruse the discussions in Pernicka (2014) and Radivojević *et al.* (2019), and the cited literature on the subject.

The use of LI data to characterize and discriminate ore sources has some advantages, mostly related to the frequent homogeneity, reproducibility, and well definable isotopic signal in a single ore body, and to the generally assumed absence of fractionation of the lead isotopes during the metallurgical processes (Stos-Gale and Gale 2009; Cui and Wu 2011). However, as abundantly discussed in the literature, it is conceptually evident that (1) it is not possible to discriminate two ore bodies having *the same geological age* on the isotopic character alone, and (2) although it is possible to firmly establish that the measured LI ratios can exclude the origin of the metal from isotopically non-compatible ores, it is not possible to firmly establish origin from a specific ores unless *all other sources* can be excluded. The interpretation process, therefore, must involve comparison with all possible sources, and in most cases it should also rely on complementary chemical, geographical, geological, archaeological, or archaeo-metallurgical information (Baron *et al.* 2014; Villa 2016). Besides the intrinsic limitations of the method, the pitfalls linked with the use of limited databases and partial or biased contextual information were at the basis of the fierce discussion on the validity of LIA during the last decades (summarized in: Pollard 2009, 2011, Pollard *et al.* 2014, Gale 2009, Cattin *et al.* 2009a, Killick *et al.* 2020). It is now generally agreed that the interpretation of LI values must be geologically sound and considered in the context of all the other material evidence in order not to incur into untenable conclusions, such as the recent claim that Chinese bronzes originated in South Africa (Sun *et al.* 2016; Liu *et al.* 2018). As an example, the early interpretation of Scandinavian object data based on a limited Alps-free database resulted in several erroneous conclusions (Ling *et al.* 2014), which were subsequently recovered by the use of a more complete reference database (Melheim *et al.* 2018; Ling *et al.* 2019; Nørgaard *et al.* 2019).

In the 80s and 90s of last century, extensive projects consistently analyzing ores, metallurgical waste, and metal objects were performed mostly by German and British teams focusing on South East-Europe and the Balkans, on the Western Mediterranean and the Aegean, on Anatolia and the Levant, most notably on the Arabah Valley. As the result, vast syntheses of ore and metal LI data

were reported for Cyprus (Gale *et al.* 1997; Stos-Gale *et al.* 1997), Bulgaria (Pernicka *et al.* 1997; Gale *et al.* 2000), or Serbia (Pernicka *et al.* 1993). These fundamental investigations resulted in the core of the existing LI reference databases of ores and objects, routinely employed during archaeo-metallurgical investigations for comparison of LI data and provenance interpretation. The most cited, publicly available, and thus widely employed LI database is certainly OXALID (oxalid.arch.ox.ac.uk/, Stos-Gale and Gale 2009), although virtually each research group built on the core data from the literature by adding LI data measured on subsets of local ores (see for example: the British Isles, Rohl and Needham 1998; the Alps, Artioli *et al.* 2016a; Scandinavia, Ling *et al.* 2013; or Iberia: Arribas and Tosdal 1994, Hunt-Ortiz 2003, Klein *et al.* 2009, Santos Zalduegui *et al.* 2004). The large amount of LIA published for the Iberian peninsula in the last decades is remarkable, especially for prehistoric copper mines with radiocarbon chronology (a compilation of available geological data can be found in Montero-Ruiz 2018). Again, the issue concerning the completeness of the reference database will not be discussed here in detail, although it is worth reminding that a careful control of the geological and mineralogical significance of the employed dataset is mandatory (Artioli *et al.* 2016a). Inquiries of available databases are generally performed by simple graphical and visual estimation of matching between the measured LI ratio on the objects and the LI reference fields of the ores. However the use of more quantitative assessments such as Euclidean distances in the 3D isotopic space (Stos 2009) and/or probability calculation by kernel density estimation (KDE: Baxter *et al.* 1997, De Ceuster and Degryse 2020) is highly recommended. Because of the large overlap in ore fields and because of the large amounts of data present in the literature, reliable provenancing will need in the future more sophisticated and flexible algorithms, possibly rooted in the rigorous geochemical and geological modeling of the time evolution of isotopic curves (Albarède *et al.* 2012; Killick *et al.* 2020). As an example, the fitting of the measured LI ratios on the objects along or across the isotopic evolution curve of the ores may give clues between different interpretation models (Angelini *et al.* 2019). Provenance analyses of course are just the starting point that should lead to the comprehension of more complex socioeconomic processes resulting in the observed metal diffusion (Rehren and Pernicka 2008; Armada *et al.* 2018; Radivojević *et al.* 2019).

In the last two decades, the number of LI investigations of ores and metal objects has substantially expanded. Many ore bodies in Europe and beyond have been actively surveyed, sampled, and characterized from the mineralogical, geochemical, and isotopic point of views. The available LI databases of ore deposits now encompass most of Continental Europe from Iberia to Poland, the British Isles, Scandinavia, most of the Mediterranean Islands such as Balearic Islands, Sardinia, Crete (for most of the European and Mediterranean deposits, see the extensive reference list in Ling *et al.* 2014). Data are also available for the major deposits in the African countries facing the Mediterranean such as Morocco, Tunisia, and Egypt (see for example Skaggs *et al.* 2012). LI data are available also for areas beyond Anatolia and the Levant, such as for example the Arabian Shield, Oman, Iran, and Armenia. Asian deposits are also being extensively mapped (Hsu and Sabatini 2019). Despite the fact that specific small areas where ores are present still need systematic survey and LI characterization (for example Slovenia, Albania, Corsica), a large scale overview of European geology shows that there is a satisfactory coverage of virtually all major ore deposits. It is true that in antiquity, especially in prehistory, even small and localized ore bodies could well be exploited for the extraction of metals that are uninteresting from the point of view of modern industrial metallurgy. However, the LI signature of most large-scale geological structures are known and available, so that during the interpretation of the isotopic archaeo-metallurgical data it is possible in many cases to predict (from the model

age of the formation) or assume (from the geological nature of the body) the likely isotopic character of the unsampled or unknown ores (Albarède *et al.* 2012). The issue is particularly relevant in the discussion of the tin metal circulation in the Bronze Age Mediterranean Sea.

No further methodological issue will be discussed here, other than refreshing the recommendation of the use of a robust database (i.e. geologically screened) when interpreting LI data. In the following discussion separate ore databases containing LI data for Cu-containing minerals, Pb-Ag containing minerals (essentially galena and argentiferous galenas) and Sn-containing minerals will be used for each specific application/interpretation. Furthermore, it is advised to use 3D LI data referred to ^{204}Pb (i.e. the use of $^{206}\text{Pb}/^{204}\text{Pb}$, $^{207}\text{Pb}/^{204}\text{Pb}$, $^{208}\text{Pb}/^{204}\text{Pb}$ ratios as plotting parameters) because they are consistent with the geological interpretation and modeling, and more discriminating between the different ore sources (Killick *et al.* 2020). Some authors even propose to use geological/geochemical parameters in place of the measured isotopic ratios (i.e. μ , related to the $^{238}\text{U}/^{204}\text{Pb}$ content in the source; κ , related to the $^{232}\text{Th}/^{238}\text{U}$ ratio; and T , the time of formation of the deposit; Albarède *et al.* 2012), although the T - μ - κ representations might be hard to be accepted in the archaeological literature. Although complex geological histories may prove difficult to model, especially when secondary processes involving input of radiogenic components are active, it has been shown in practice that the expert application of model ages to ore deposits may be an efficient way to discriminate metal sources (Desauty *et al.* 2011), because the age model relies on objective geological parameters concerning the common Pb composition in the mantle and the $^{238}\text{U}/^{204}\text{Pb}$ composition in the crust (Stacey and Kramers 1975; Albarède and Juteau 1984).

Archaeology is essentially predicated on the observation of change and difference, either in time or space. Accordingly, this review will focus on the available evidence for metals and metallurgy in the Central Mediterranean, attempting a broad interpretation of the published LI data in terms of (1) the chronology of metal diffusion in the area, and (2) the possible sources exploited at different times, in relationship with the proposed models of the rise and diffusion of metallurgy.

Because archaeological chronology may be very different even for geographically contiguous areas, we will rely on a simplified time table based on absolute dates and referred to the Italian chronology, as central in the Mediterranean context (Tab. 1), although of course it is hardly applicable to all local archaeological sequences. For example the period roughly related to the Beaker event (appx 2700–2200 BC, Olalde *et al.* 2018) corresponds to the Late Neolithic in Southern France and Switzerland, the Late Eneolithic (or Late Copper Age, see: Pearce 2019) in Northern Italy and in most of the Islands (Corsica, Sardinia, Sicily), and the Middle Bronze Age in several of the Balkan countries. Obviously societal and cultural changes reflected in the archaeological record take place at different times and with different paces in geographically distinct areas, and metallurgy is not immune from this general rule (Diamond 1997; Pare 2000; Roberts and Thornton 2014; Armada *et al.* 2018).

COPPER AND COPPER ALLOYS

Concerning the wider picture of the rise of copper metallurgy, there are two major lines of thinking (Pearce 2015; Montero-Ruiz and Murillo-Barroso 2017). The first one is based on the diffusionist paradigm following Childe and Wertime, arguing for a single origin of the metallurgical knowledge and technology in the Near East or Anatolia (Roberts *et al.* 2009). The second one favors Renfrew's idea (Renfrew 1969) of multiple centers of copper metallurgy, located in the Near East (Iran, Tal-i Iblis, late 6th millennium BC: Caldwell 1968, Pigott and Lechtman

Table 1 Approximate local chronology of appearance of metals. In italics are reported some of the important metal-bearing cultures cited in the text

Age BC	Iberia	Southern France	Italy	Balkans	Greece	Aegean - Crete
c. 5000–				Early Chalcolithic [<i>Vinča</i>]		
4500				Middle Chalcolithic [<i>Varna</i> , <i>Gumelnița–Karanovo</i>]		
c. 4500–			Middle-Late Neolithic [<i>VBQ</i>]	Late Chalcolithic–Proto		
4000			Late Neolithic [<i>Late VBQ</i> , <i>Chassey, Lagozza</i>]	Bronze Age		
c. 4000–			Early Eneolithic [<i>Rinaldone</i> , <i>Gaudio, Remedello I</i>]	Early Bronze Age	Early	Early Minoan
c. 3500–	Early Chalcolithic		Middle-Late Eneolithic	Early Bronze Age	Helladic	Early Minoan
3000	Middle Chalcolithic		[<i>Remedello II</i>]	Early Bronze Age	Early	Early Minoan
c. 3000–			Late Eneolithic-[<i>Campaniforme</i>]	Middle Bronze Age	Helladic	Early– Middle
2500		Late Neolithic [<i>Beaker</i>]	Early Bronze Age	Late Bronze Age	Early	Minoan
c. 2500–	Late Chalcolithic - Early Bronze Age [<i>Beaker</i>]		Early–Middle Bronze Age	Late Bronze Age	Middle	Middle
2000	Early Bronze Age [<i>El Argar</i>]	Early Bronze Age	Middle-Late Bronze Age	Late Bronze Age–Iron Age	Helladic	Minoan
c. 2000–					Late	Late Minoan
1500	Middle – Late Bronze Age	Middle Bronze Age [<i>Timulus - Urnfield</i>]			Helladic	
c. 1500–					Late	
1000					Helladic	

2003), the Balkans (Serbia, Belovode, 5000–4650 BC: Radivojević *et al.* 2010), and Iberia (Spain, Cerro Virtud, 4900–4450 BC: Ruiz-Taboada and Montero-Ruiz 1999) because of the early dates of archaeological evidence. With no claim to resolve the issue, we should keep these models in mind when interpreting the LI data available for the Central Mediterranean area. The selected focus on the Central Mediterranean (Italy, the Mediterranean Islands in the Tyrrhenian area, and the neighbouring regions) wishes to investigate the role of this region as a key hub involved in different, and sometimes multiple, cultural and economic exchanges. The subtle interplay among the socio-economical frame, the technological knowledge, and the exploitation of different sources of metal around the Mediterranean at different times is still there to be deciphered.

One of the problems in decoding the rise and diffusion of metallurgy in the broad Central Mediterranean region is that the already scarce archaeological evidence has been poorly characterized by modern archaeometric tools, and part of the information is sometimes flawed by misinterpretation. For example, if we examine the early metallurgical evidences often reported for the early fourth millennium, we can find systematic citations of Lipari (Acropolis: Bernabò Brea and Cavalier 1980) as evidence of the presence of copper slags (see for example: Cocchi Genick 1994, Pessina and Tiné 2008, Dolfini 2014a), and Grotta della Monaca (Calabria: Larocca 2005, Quarta *et al.* 2013) as copper mine. As a matter of fact, the Lipari slag has been recently analysed and found to be the product of pyrotechnology unrelated to metallurgy (Martinelli *et al.* 2016), and the Calabrian cave was constantly used in prehistoric times to obtain iron and copper pigments (Larocca 2010), though there is no evidence of extractive metallurgy. Reporting these sites in the maps of early copper metallurgy severely overemphasizes its importance in the South of Italy and Sicily, which are remarkably devoid of copper resources from the geological point of view. Similarly, the most promising evidence of primary or secondary copper metallurgy (slag, raw metal, technical ceramics) in Central and North Italy (Botteghino di Parma: Maffi *et al.* 2014) has not been fully analysed. In the late fifth millennium and early fourth millennium most of the metal circulating in Northern Italy is in the form of awls, pin, wires, besides a number of copper axes, mainly skeuomorphs of polished stones axes (Pearce 2007, 2015). Although there are yet no LIA of the axes, several copper awls and pins of the fourth millennium from Northern Italy have been isotopically characterized (Fig. 1). Invariably, all objects are made of

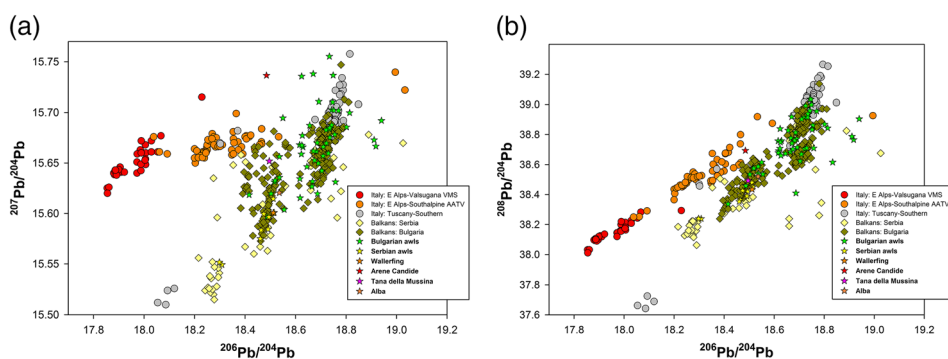


Figure 1 LI ratios measured for Northern Italian awls and pins of the fourth millennium BC: Arene Candide (Campana *et al.* 1996), Tana della Mussina (Tirabassi 2013; Canovaro *et al.* 2020), Alba (Venturino Gambari 2002; Angelini *et al.* 2020). The data are compared with broadly coeval materials from literature: the awl from Wallerfing, Germany (Höppner *et al.* 2005), Bulgarian awls, pins and borers (Pernicka *et al.* 1997; Gale *et al.* 2000), Serbian awls and borers (Pernicka *et al.* 1993). [Colour figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.com)]

Balkan copper, except maybe the punch from the Arene Candide cave (Campana *et al.* 1996), which will be discussed below in detail. Comparison of the Northern Italian awls with the Serbian and Bulgarian awls, pins, and borers available in the literature (Pernicka *et al.* 1993; Pernicka *et al.* 1997; Gale *et al.* 2000) clearly shows that the Balkan deposits were already heavily exploited. Balkan copper was thus diffusing in the whole of Northern Italy, as shown by the LI data measured on the available awls of the fourth millennium found in Alba (Venturino Gambari 2002; Angelini *et al.* 2020) and Tana della Mussina (Tirabassi 2013; Canovaro *et al.* 2020). The LI data distribution (Fig. 1a,b) clearly indicates that no copper from the South-Eastern Alps or from Tuscany was yet circulating in Northern Italy in this early period. Possibly this hypothesis could be extended to the Alpine area, based on the LI data measured on the German awl found in Wallerfing (Höppner *et al.* 2005), which is also made of Balkan copper. The LI data reported for the awl found at the Arene Candide cave, Western Liguria (Campana *et al.* 1996), is anomalous. It was tentatively interpreted to be compatible with Alpine or Swiss ore deposits, but actually the LI data do not really fit with any known copper deposit present in the database and only have a mild isotopic affinity with some Catalan deposit. On one hand, the accuracy of these old TIMS data should be checked with modern multi-collector techniques, because ^{204}Pb could be rather hard to measure, especially in early eneolithic copper samples, which are frequently very poor in lead (see for example Artioli *et al.* 2017). This is exactly the case of the Arene Candide punch that is reported to have no lead within the sensitivity limits of X-ray fluorescence spectrometry (Stos-Gale in Campana *et al.* 1996). On the other hand, some of the Bulgarian awls carry an isotopic signal extending the Bulgarian ore field towards higher $^{206}\text{Pb}/^{204}\text{Pb}$ and $^{207}\text{Pb}/^{204}\text{Pb}$ values (Fig. 1), so it is also possible that some radiogenic Balkan ores are missing from the database. The issue clearly needs more data to be resolved.

After or in parallel with this early phase of introduction of metal objects made with Balkan copper, there is evidence of a period of intense metalwork whose center of gravity is Central Italy (Dolfini 2014a; Dolfini 2014b). The objects are mostly related to the *Rinaldone* culture but extending southward and northward with the *Gaudo*, and *Remedello I* cultures respectively (Tab. 1). Based on the metal object occurrences, the rise and diffusion of this metallurgical phase is arguably dated to the mid fourth millennium BC (Dolfini 2013), though the earliest fully investigated evidence of developed extraction and reducing metallurgy in Italy is the site of S. Carlo, near Piombino, Tuscany (Artioli *et al.* 2016b), which has been radiocarbon dated to about 3200 BC. Interestingly, the site shows a remarkably developed reduction technology leading to the complete melting of the slag and a good efficiency of reduction and metal recovery. The technique is much more advanced with respect to what is reported elsewhere for “immature” chalcolithic smelting technology (Bourgarit 2007). The LI investigation of the copper droplets present in the S. Carlo metallurgical set of course match the signal of the nearby ores of the Temperino Mine and surrounding mineralizations of the Tuscan domain (Dini and Boschi 2017). However more importantly they match closely the LI signal of the coeval Iceman copper axe from Hauslabloch, Alto Adige (Artioli *et al.* 2017), and the axe from Zug-Riedmatt, Switzerland (Gross *et al.* 2017). These results indicate that: (1) there is a developed copper metallurgy in the Tyrrhenian coast of Central Italy in the last part of the fourth millennium BC, and (2) the produced copper was moving as far North as Switzerland and Tyrol, although these finds represent at present the Northern most limit of diffusion.

Interestingly enough, the LI signal of the long-studied Ligurian mines (Libiola and Monte Loreto: Campana *et al.* 1996, Maggi and Pearce 2005, Pearce 2007, Nimis *et al.* 2017) is very different from that of the Tuscan, West-Alpine, and French Massif Central ores (Artioli *et al.* 2013)

and therefore easily recognizable. However, to date no measured object can be referred to the Eastern Ligurian mines.

A LI signal compatible with Aegean ores has been reported for a flanged axe of Levantine typology in the Rinaldone context from Casetta Mistici (Anzidei *et al.* 2018) so that the object should be considered an exotica. However, it should be remarked that the full isotopic data have not been published, and no complete statistical analysis can be performed on the partially reported LI ratios in order to confirm the suggested interpretation. Furthermore, the typology is well known from the Levant, but there are no dated occurrences before the middle of the third millennium BC, so its presence in a Rinaldone context is surprising and should be confirmed. On the other hand, for the most part the copper metal forming the typical Rinaldone objects is clearly compatible with the Tuscan ores (Dolfini *et al.* 2020), both in terms of Pb isotopes and chemical composition. Interestingly, a few of the analysed objects from Central Italy are isotopically incompatible with the Tuscan signal, showing affinity with the ore deposits of the Western Alps and even the ores of the French Massif Central. The data therefore indicate a remarkable dynamics in the metal flow in the second part of the fourth millennium, indicating the arrival of metalwork in Tuscany through what can be considered a “Tyrrhenian” circuit encompassing Southern France, the Western Alps, Liguria, and Tuscany. This hypothesis may find support in existing cultural links, such as the geographical distribution of the statue-menhir, which are present in Southern France and Italy, and broadly fits proposed models of metallurgical diffusion in the Central Mediterranean (Dolfini 2013). It would be extremely interesting to investigate whether Sardinia and Corsica were included in this metal circuit (Melis 2014; Pearce 2018), because there is evidence that Sardinian silver was reaching the Italian Peninsula already in the early fourth millennium (Venturino *et al.* 2018). Ongoing projects based on LI are aimed to better define the area of diffusion of the Tuscan metal in the second half of the fourth millennium BC. The analyses of the metal objects of well dated sites, such as the Remedello cemetery, Reggio Emilia (Remedello Phase 1, De Marinis 2013), and Celletta dei Passeri, Forlì (Miari *et al.* 2017), among others, will be fundamental in clarifying the issue.

The situation is totally different if we look at LI signal of similar objects (awls, pins, rods) dated to the third millennium BC (Fig. 2). All of the analysed objects from Northern Italy and the Alpine region are made of South-Eastern Alpine copper, and comparison to the available data on coeval awls from Switzerland (Saint-Blaise/Bains des Dames site: Cattin *et al.* 2009b) show

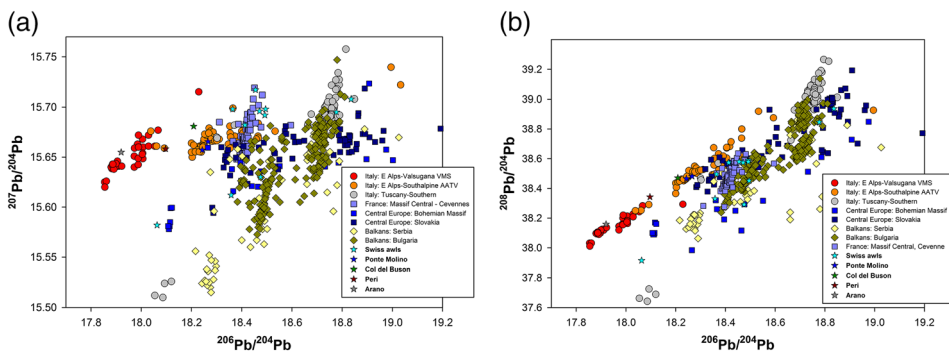


Figure 2 LI ratio measured for Northern Italian awls and pins of the 3rd millennium BC: Ponte Molino (Longhi and Tirabassi 2019), Col del Buson (Angelini *et al.* 2011; Artioli *et al.* 2013), Peri (Angelini 2004), Arano (Pernicka and Salzani 2011). The data are compared with Late Neolithic awls and rods from Switzerland (Cattin *et al.* 2009b). [Colour figure can be viewed at wileyonlinelibrary.com]

that the Swiss objects are made of copper from the several different sources that were active at time, including the French Massif Central, Bohemia, and Slovakia. The fact that all analysed Italian objects are made of South Alpine copper indicates that the systematic and massive exploitation of the South-Eastern Alpine chalcopyrite ores is essentially coincident with or slightly preceding the Beaker event (in Italian *Cultura Campaniforme*, Tab. 1). This is supported by the evidence derived from the distribution and large amount of copper smelting slags found in the Trentino and Alto-Adige area, mainly in the Valsugana Valley, the Adige Valley, and Isarco Valley (Artioli *et al.* 2015). All of the slag sites are dated to just before or coinciding with the Beaker event, and most of them were still active in the transition to the Early Bronze Age. Not surprisingly, virtually all the analyzed Northern Italian objects of the South-Eastern Alpine copper (see Fig. 6 and Table 3 of Artioli *et al.* 2016a).

Based on the absence of slag or known metallurgical sites, in the Southern Alpine region there seems to be apparently a negligible or even no copper production during the Early Bronze Age (EBA). In the Aegean, there is apparently a similar net decrease in copper production during the second millennium. In the words of Bassiakos and Tselios (2012): “Several sites located in various islands of the Aegean, such as Thassos, Kythnos, Seriphos, Siphnos, Parapola and others, proven (archaeologically and technologically) to be third millennium copper production centres exploiting local ore-sources, appear to be inoperative, with few exceptions, during the 2nd millennium”. However, if the available data for EBA objects found around the Lake of Garda (Pernicka and Salzani 2011) are plotted (Fig. 3), the LI signal shows a South Eastern Alpine signature for most of them. The data were not interpreted at the time of publication, because by then the data for South Alpine ores (Nimis *et al.* 2012) had not yet been published. The interesting aspect is that in the EBA object we start observing a chemical and isotopic complexity that was not present in Eneolithic objects. On one side the objects made of pure copper or arsenical copper bear the typical signature of the Valsugana ores (Calceranica, Vetriolo). The diffuse presence of arsenopyrite in the Valsugana ore district is fully consistent with the indication derived from the isotopic ratios. However, the isotopic signal of the objects made of typical falhore-derived copper (i.e. containing Ag, Sb, As, Ni) is also compatible with the Southern Alpine ores (Fig. 3), whereas there is minimal compatibility with the typical falhores of the Inn Valley in Tyrol (Höppner *et al.* 2005). This is surprising because there are few falherz-type ores in the Southern Alps (notably the tetrahedrite-based deposits in Carnia (yellow circles in Fig. 3), including Monte Avanza, and the Montagiù deposit in Trentino, Nimis *et al.* 2012). For all of them there is no

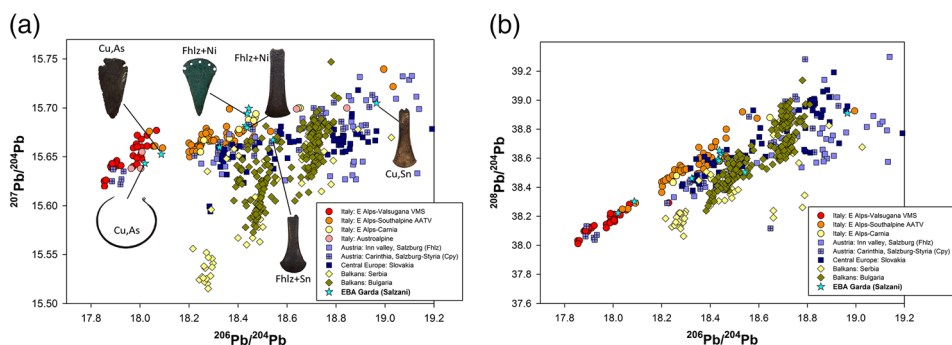


Figure 3 Plot of LI data of EBA objects from Pernicka and Salzani (2011). Figure of the objects and major elements composition are from the same volume: Cu,As, arsenical bronze; Cu,Sn, tin bronze; Fhlz+Ni, Ni-containing falherz copper; Fhlz+Sn, Sn-containing falherz copper. [Colour figure can be viewed at wileyonlinelibrary.com]

evidence of prehistoric exploitation to date. Bearing in mind that absence of evidence is not evidence of absence, the simplest way to explain both the measured isotopic and chemical signals is either to assume an early extraction of the Southern Alpine falhore deposits, such as the Monte Avanza one, or to invoke a mixing between the Valsugana chalcopyrite-arsenopyrite derived copper and the falherz derived copper, probably from Slovakia or Tyrol. In either case, the LI measurements are showing an unexpected dynamics and complexity in EBA copper metallurgy. This chemical complexity remains a constant in the Eastern Alpine area for most of the Bronze Age.

The systematic lack of copper smelting slag in the Alpine area is even more evident for the Middle and Recent Bronze Age (approximately 1600–1300 BC), a period in which the massively produced Southern Alpine copper is flowing to the plains South of the Alps and also toward Scandinavia (Ling *et al.* 2019). The apparent lack of smelting evidence compared to the massive amount of copper objects dated to MBA-RBA could be substantially reduced by recent new chronological evidence of the known slag heaps in the Southern Alps (Pearce *et al.* 2019). Although it is not the focus of this paper, it is worth reminding that the recent results stemming out of LI investigations are gradually contributing to change our picture of the copper and bronze trades in Continental Europe (Radivojević *et al.* 2019), specifically showing a rather unexpected Southern Alpine origin for most of the Scandinavian copper. Despite the scarcity of LI data for the Central Mediterranean Bronze Age, there is a growing evidence that Alpine copper was flowing southward to the Venetian plain and the Po Valley (see for example: Cupitò *et al.* 2015; Vicenzutto *et al.* 2015; Angelini *et al.* 2015a), toward East into Friuli Venezia Giulia (Canovaro *et al.* 2019), and even found its way down to the metal workshops of Southern Italy and possibly to Greece and the Eastern Mediterranean (Jung *et al.* 2011). The copper flow from the Southern Alps intensifies again in the Late Bronze Age (LBA), related to the pervasive presence of smelting slag linked to the Laugen/Luco culture (Cierny 2008; Addis *et al.* 2016, 2017; Pearce *et al.* 2019). Once more the LI data help in drawing a clear picture of the territorial exploitations of the mines and the links between the mines and the smelting sites (Addis 2013). Of course, in the LBA Northern Italy is flooded by Alpine copper, as testified by the nature of the Frattesina metal (Angelini *et al.* 2015b; Villa and Giardino 2019). We fully agree with the vision of Pearce *et al.* (2019) that in the LBA Frattesina is “an important trans-shipment node between continental Europe and the Mediterranean and a manufacturing site at the centre of the metals trade ... rather than subsidiary to Etruria mineraria”.

If we focus further South, along the East–West trading routes, the LBA copper production seems to be dominated by Cyprus. All oxhide ingots found around the Mediterranean (Cyprus, Crete, Sardinia) and dated between the 14th and the 11th century BC are consistent with the known field of Cypriot ores (Fig. 4). They include the investigated ingots from the renowned Uluburun and Cape Gelidonya shipwrecks, dated to about 1300 BC and 1200 BC, respectively (Gale and Stos-Gale 1986, 2005; Stos-Gale *et al.* 1997; Gale 1999, 2005, 2011; Stos 2009). It is clear that in the Late Bronze Age, copper from Cyprus was massively produced and traded in the Western Mediterranean and diffused as far as Sardinia, the North coast of the Black sea, and Anatolia (see details in: Lo Schiavo *et al.* 2009, Kassianidou 2013), so that by the 13th century BC, Cyprus was certainly the main copper producer and exporter throughout the Mediterranean sea. By the use of Cu isotopes it has also been proposed that copper production in Cyprus shifted from oxidic ores to chalcopyrite (Jansen *et al.* 2018), a commonly held assumption that is not possible to prove with Pb isotopes alone. Thanks to the distribution of the ingots, the literary sources, and the amazing discovery

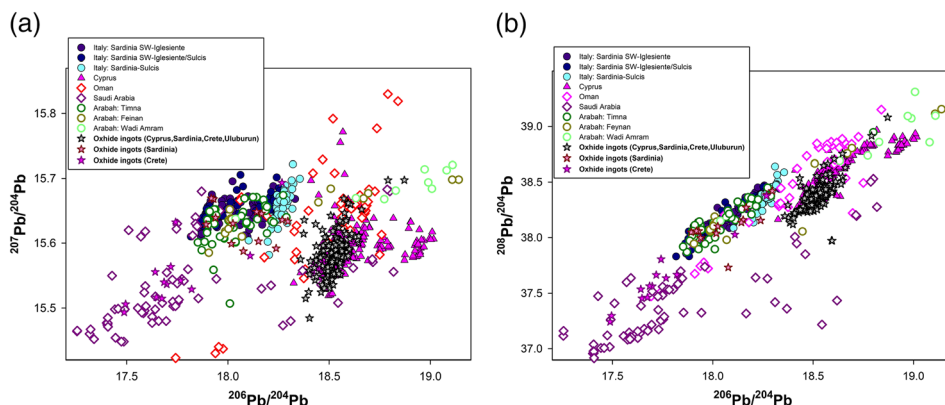


Figure 4 Measured LI ratio for LBA oxhide ingots from Cyprus, Sardinia, Crete, and the Uluburun and Cape Gelidonya shipwrecks (see text for details). [Colour figure can be viewed at wileyonlinelibrary.com]

of Bronze Age shipwrecks, it is possible to start picturing the fascinating and grand metal trade in Bronze Age Mediterranean. Interestingly, similarly to what happens in the South-Eastern Alps, in LBA the place that originates most of the traded copper is surprisingly devoid of local metalwork (Papasavvas 2012), as if production and consumption were neatly disjointed. This may be a warning that simple models assuming provenance from the mere density in the geographical distribution of finds can be deceiving.

Despite the fact that copper started to be produced in Cyprus on a small scale as early as the 17th century BC (Gale 1989), some of the earliest oxhide ingots found in Crete, especially those from Hagia Triada, and dated to the Late Minoan IB period (appx. 1500–1450 BC) are not compatible with the Cypriot isotopic signal (Fig. 4, data from Table 22.2 of Stos-Gale 2011). As Noel Gale clearly stated following the early investigations: “At any rate, Cypriot oxhide ingots were not coming to Crete in the period 1550–1450 B.C.; indeed Cypriot copper was almost certainly not being cast into oxhide ingots as early as this. In this period oxhide ingots were made elsewhere, though we know from our work on the Middle Cypriot site Alambra that Cypriot copper was being used on a small scale as early as about 1800 B.C.” (Gale 1989). Actually some of the Late Minoan IB oxhide ingots in Crete indeed do carry the Cypriot isotopic signal (Stos-Gale 2011), testifying a dynamic metal trade from various sources. However, the origin of the non-Cypriot copper in Crete has been the source of hot debate because of the consequences on the interpretation of the Late Bronze Age copper trade. The very low values of the measured LI ratio are compatible with rather old deposits, incompatible with most of the Mediterranean ores including the Cambrian-hosted Pb–Zn deposits of Sardinia (Boni and Koeppl 1985) but compatible with the Precambrian ores in Eastern Egypt, the Sinai and the Arabian Peninsula, including Oman. Interestingly, some of the recently analysed copper finds from Funtana Cubierta, Sardinia, dated to the 13th century BC (Montero-Ruiz *et al.* 2018), including several fragments of oxhide ingots, and a few of the Sardinian objects analyzed by Begemann *et al.* (2001) all carry a non-Cypriot isotopic signal compatible with the Eastern deposits, mainly those of the Arabah valley. Despite the substantial overlap between the LI values of the Sardinian and Arabah ores (Fig. 4), the latter are preferred on the basis of closer Euclidean distances and trace element patterns (Montero-Ruiz *et al.* 2018). The fact that the Crete oxhide ingots carry the Arabian shield LI

signature and that the Sardinian oxhide ingots carry the Arabah LI signal is once more an indication of the previously unsuspected dynamics of the ancient Mediterranean copper trade.

LEAD

LI are, of course, well-suited to decode the provenance of lead objects (Baron *et al.* 2006; Cattin *et al.* 2009b), provided that metal from different sources is not mixed. As an example, we gathered most of the available LI data on investigated Roman lead ingots from shipwrecks around the Central Mediterranean (Fig. 5).

The plot of the lead ingots LI data is instructive, and much information can be derived:

- 1 It can be straightforwardly appreciated that the isotopic plots using the data relative to ^{204}Pb can discriminate very well among the major sources of Pb around the Mediterranean exploited at the peak of the Roman Empire (Domergue 1990; Hirt 2010), here the major lead ore deposits of Sardinia, Iberia, France, and Greece are reported.
- 2 The investigated Pb ingots recovered around the Western and Central Mediterranean (see legend of Fig. 5) indicate that the Carthago Nova district in Iberian Murcia was the major source of lead at the time represented by the shipwrecks (between the end of the second century BC and the middle of the first century AD). In this period the British, German, and Balkan deposits were not yet under the control of Rome. The data reported for two shipwrecks of the third century BC (Cabrera 2, from the Balearics and Îlot de Brescou at Agde, Trincherini *et al.* 2009) indicate that the coastal route for transport of the Cartagena lead was already in use during Republican times. The data also seem to suggest that some lead was originating in the Linares/Jaen area (Cabrera 5 wreck), the French Massif Central (Saint Maries de la Mer wreck), and negligible lead was produced in Sardinia. Although it is hard to have a precise chronology, the LI data obtained from ship artefacts recovered in South Western Sardinia (anchors, rings, bars: Begemann and Schmitt-Strecker 2001) denote a rather more complex dynamics of metal flow. In fact, the various parts of the anchors indicate lead production in

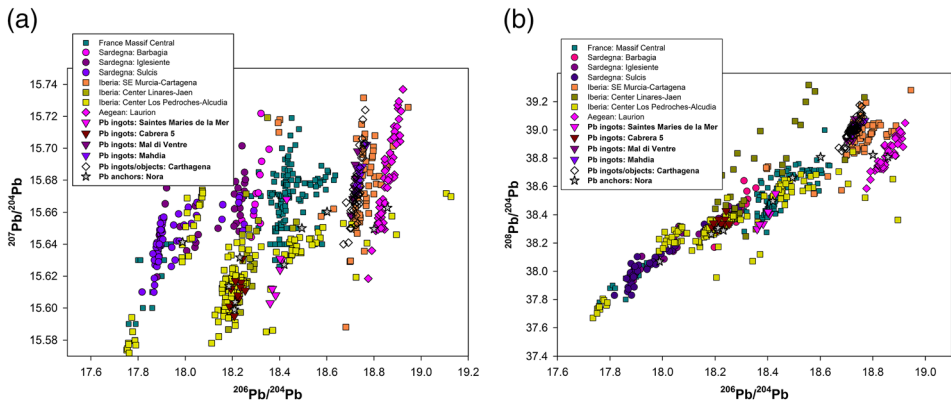


Figure 5 LI data of available measurements on Roman lead ingots from shipwrecks in the Western and Central Mediterranean: Saint Maries de la Mer and Cabrera 5 (Trincherini *et al.* 2001), Mal di Ventre (Pinarelli *et al.* 1995), Mahdia (Begemann and Schmitt-Strecker 1994). The data for Carthago Nova (Trincherini *et al.* 2009) include ingots from several shipwrecks (Cabrera 2, Escombreras 2) plus ingots from the Murcia mines and several other inland sites. The anchor data (Begemann and Schmitt-Strecker 2001) include various Pb components of anchors and ships recovered underwater in South-Western Sardinia. [Colour figure can be viewed at wileyonlinelibrary.com]

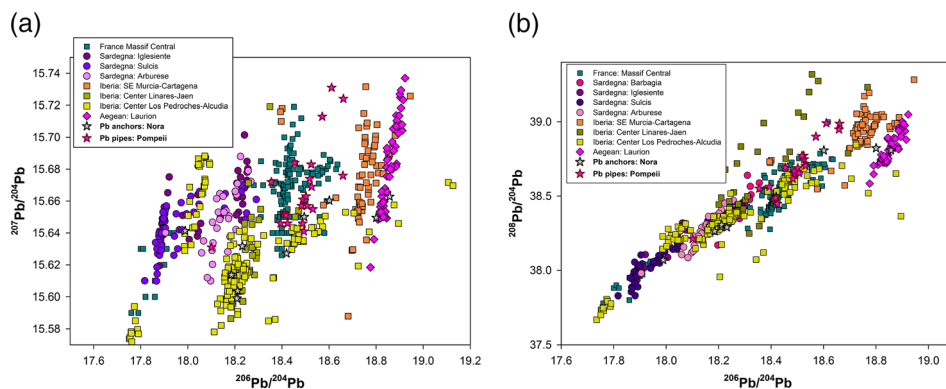


Figure 6 LI data of available measurements on Roman lead pipes from Pompeii (Boni *et al.* 2000) and some implements and anchors from ships (Begemann and Schmitt-Strecker 2001) recovered underwater in South-West Sardinia. [Colour figure can be viewed at wileyonlinelibrary.com]

Sardinia (Sulcis area), Iberia (Valle de Alcudia), the French Massif Central, and Greece (Laurion)(Fig. 5).

- The LI data for the Saint Maries de la Mer ingots are nicely following the trend line of the French Massif Central and actually extend the Pb-ore field to lower $^{207}\text{Pb}/^{204}\text{Pb}$ values, thus hinting that ore data for several important mine districts are missing from the available database of French Pb ores (Baron *et al.* 2006).

There is one further complication in the interpretation of the LI data of Pb artefacts. The issue is well exemplified by plotting the data obtained on several samples of the great hydraulic network of water pipes built under Augustus in Pompeii (Fig. 6, data from Boni *et al.* 2000). Apart from one object (97P11B, that is a segment of lead pipe from Casa di Orfeo) which is fully compatible with the Early Paleozoic lead ores from the Sardinian Sulcis area, most of the LI data plot in a field severely overlapping the isotopic field of the French Massif Central ores (Fig. 6). They also broadly follow a similar evolution line. Following the originally published interpretation, which however omitted the French ores from the discussion, these data have been constantly interpreted as a mix of Sardinia-, Iberia-, and Laurion-derived Pb metal. The samples therefore are “the results of repeated re-meltings and mixing and represent an integration, over a period of several centuries, of the manufacture of lead objects around Pompeii” (Boni *et al.* 2000). Far from challenging this interpretation, here we just cast a warning about a simplistic interpretation of LI data of Roman lead objects. If we exclude the French origin (which is however plausible given the signal found in the Sainte Maries de la Mer ingots) and use simple mixing lines (Bode *et al.* 2009; Villa 2016) to assess the presence of the other sources as defined by the isotopic fields of the ingot data, it is clear that Laurion- and Central Iberian-derived lead is minimal, and most of the Pompeii pipes are made of lead from *Carthago Nova*, slightly diluted by Sardinian lead.

SILVER

Apart from the ores in Rio-Tinto, Iberia, where silver is present in secondary jarosite (Rothenberg and Blanco-Freijeiro 1982; Domergue 1990; Anguilano *et al.* 2010) and scarce occurrences in the native form (such as in Sardinia, in association with silver chlorides), most silver in ancient

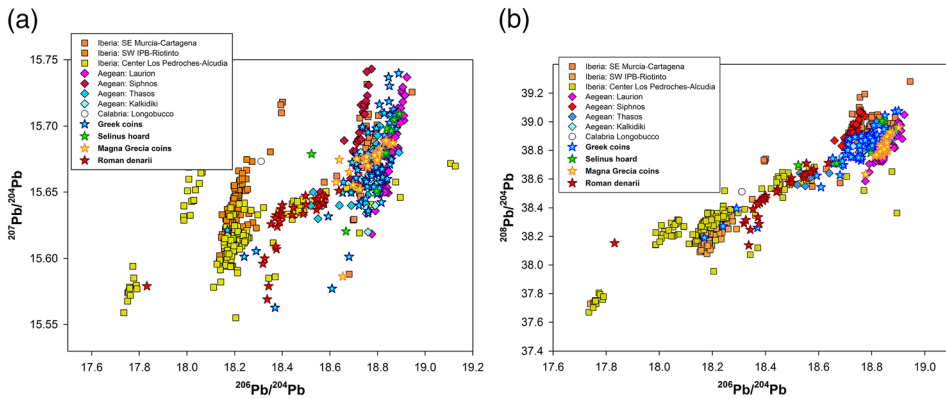


Figure 7 LI data of available measurements on ancient Greek coins (6th–4th centuries BC, from OXALID, see references in Stos-Gale and Davis 2018), the Selinus hoard (Beer-Tobey et al. 1998), Magna Grecia coins (Birch et al. 2018), and Roman denarii of Augustan age (Ponting et al. 2003; Butcher and Ponting 2005). [Colour figure can be viewed at wileyonlinelibrary.com]

times was derived from mixed argentiferous lead ores by cupellation processes (Nriagu 1985; Bachmann 1993; Meyers 2003). This is positive news, because if the mineral is rich in lead (i.e. galena), then the reduced silver metal should have the same LI signal as the ore, therefore being a reliable tracer for provenance. However, in the case of Pb-poor jarosite, the Pb content of the mineral may not be sufficient to induce an efficient redox process, and external lead must be added to the mixture. This is exactly the case of the Rio Tinto-derived silver, where lead from Carthago Nova was imported to supply the cupellation process (Anguilano et al. 2010). Furthermore, cupellation was also used to refine silver from debased metal (Rehren and Kraus 1999). Actually, the control of the metal purity and the silver content in coins requires its purification by cupellation, leading to foreign lead contamination. These issues, compounded to the problem of re-melting of coins (Guénette-Beck et al. 2009), should be kept in mind when dealing with the LIA of ancient silver.

Despite these problems, LI analysis has been widely used, especially for ancient coins. As an example, Fig. 7 reports some of the published data for ancient greek coins (sixth–fourth centuries BC), both from the Eastern Mediterranean (Stos-Gale and Davis 2018) and from the Greek colonies in Southern Italy (*Magna Grecia*, Birch et al. 2018), and for Roman denarii of Augustan age (first century AD: Ponting et al. 2003; Butcher and Ponting 2005).

Concerning ancient Greek coins, “virtually all the silver used to mint coins in the Archaic period in the Western Mediterranean came from the Greek motherland and its immediate surroundings” (Birch et al. 2018). Not surprisingly, the recent re-evaluation of the extensive dataset on Eastern Mediterranean archaic coins gathered by the OXALID project (Stos-Gale and Davis 2018) clearly indicates that both the mines located in continental Greece (Laurion, Trakia, the Rodhopes) and also some of the other mines located in the Islands (Siphnos, Thasos) were the source of silver for minting. A little more surprising is to observe that also the coins of the Greek colonies in Southern Italy were also made of Eastern Mediterranean silver (Fig. 7, see the LI data for the *Magna Grecia* coins and the Selinus hoard). “Thus, Greek poleis in Southern Italy and Sicily in the period up to approximately 470 BC were definitely not using locally mined silver, nor were they tapping into Carthaginian or Etruscan sources” (Birch et al. 2018). As confirmation, and an addition to the published data, we present now a new measurement (white circle

in Fig. 7) of the only Ag-containing mineral assemblage present in Calabria, the one relative to the sphalerite-argentiferous galena of Longobucco, known to be exploited for silver in Medieval times (Adorisio 2015). Apparently none of the investigated Magna Grecia coins were made of the Longobucco ores, not even those minted in Sybaris, which is located only few tens of km away from the mine.

There are however a few Greek coins falling outside the Eastern Mediterranean ore fields. Interestingly enough they are quite compatible with some of the Iberian ores, including the jarosite-based ores of Rio Tinto and the mines around the Valle de Alcludia (yellow squares in Fig. 7) that are indicated as the possible source of Argaric silver (Murillo-Barroso 2013). In fact, systematic survey of recent LI data seems to support the continuity of silver extraction in Iberia from the Early Bronze Age of El Argar until the Early Iron Age, so that the early indigenous mining seem to slowly give way to the later Phoenician control and technology, with introduction of cupellation (Murillo-Barroso *et al.* 2016). In this context, it is not impossible that some Iberian silver was transferred to the Eastern Mediterranean, as inferred by recent investigations of Phoenician silver (Eshel *et al.* 2019; Wood *et al.* 2019).

Concerning Roman denarii, their provenance is still problematic. Most of the measured data lie in a well-defined isotopic field corresponding both to Iberian ores, and to the mixing line joining the Rio Tinto and Cartagena ore fields. The hypothesis that they were made of Iberian silver, or that the silver was refined using the already mixed lead found in Pompeii (see discussion above and Fig. 6) for cupellation are both suitable. The solution seems more direct than the original proposal of the use of British or German lead for the cupellation of the Iberian metal (Ponting *et al.* 2003, Butcher and Ponting 2005). However, a general overview of the LI data for late Roman silver (Fig. 8) including Late Roman and Gallo-Roman hoards (third–fourth centuries AD) and the cited Roman denarii (1st century AD) share several features. (1) None of the circulating silver carries a homogeneous LI signal compatible with a single ore district. (2) The hoard and coin data as a whole plot in a field streaming between the Iberian and East Mediterranean ores, and overlap with the field of the Pompeii lead pipes. Therefore, the interpretation of the lead pipes of Pompeii as a mix of lead with different provenance may as well be applicable to the silver objects. (3) Assuming a simple mixing model of two silver sources (or the equivalent refining from a mixed lead metal), it is relatively simple to account for the composition of the Marengo hoard and most of

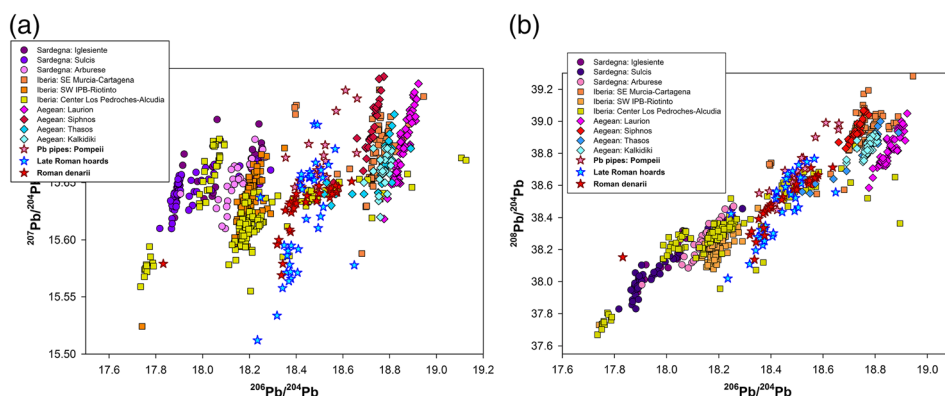


Figure 8 LI data measured on Late Roman silver objects, compared with the data of the cited Roman lead pipe measurements (Boni *et al.* 2000). The Roman denarii data are as in Fig. 7. The Late Roman silver hoards are Marengo (Angelini *et al.* 2019), Boscoreale (Berthoud *et al.* 1989), Berthouville, Notre Dame d'Allencon, Graincourt-lès-Havrincourt (Baratte *et al.* 1985). [Colour figure can be viewed at wileyonlinelibrary.com]

the Roman denarii using Sardinia and/or Rio Tinto as source A, and Cartagena and/or Greek deposits as source B. However, several of the Roman denarii and most of the Gallo-Roman silver hoards found in France show rather low values of the $^{207}\text{Pb}/^{204}\text{Pb}$ ratio, which can hardly be accounted for by the Mediterranean ore districts. These low values are also well outside the known field of the French Massif Central ores, even if the field is extended according to the Saint Maries de la Mer ingots (see discussion above and Fig. 5). For these samples the proposed contribution of British silver and/or British lead for cupellation refining may be a reasonable solution.

In any case, LI data of Late Roman silver objects show a complexity that is far higher than that of pre-Roman Iron Age objects, which most authors interpret without the need to invoke mixing, debasing, and complex refining processes. This is without doubt related to the rapid evolution of silver metallurgical techniques in the Iron Age, an evolution likely stimulated by the massive request for silver in complex societies, well beyond the basic aesthetic value of this shining metal. The rapid acquisition of silver of a high symbolic value (i.e. reference monetary value) is fundamental to understand the modes of production, diffusion, and finally storage as hacksilver, hoard, or treasure (Patterson 1972; Balmuth 2001).

TIN

It is generally considered that lead contained in tin to produce bronze does not significantly affect the lead signature of the copper because of the rather low amount of lead incorporated in tin minerals (mainly cassiterite) during formation. This assumption, which has not been disproved to date, has two consequences: the first is that LI may be used to provenance copper in tin bronzes, and the second is that the signal of tin is too low to be measured on alloyed metals. Therefore, if we want to use LIA to trace tin, we must measure pure tin objects, despite the fact that they are rare in the archaeological record, and we must be aware that in the past claims have been made about lead fractionation during the reduction process (Clayton 2001).

As recently as this year Berger *et al.* (2019b) state that “The origin of the tin used for the production of bronze in the Eurasian Bronze Age is still one of the mysteries in prehistoric archaeology”. However, the authors also show convincingly that the radiogenic character of cassiterite may be used to define the approximate age of the deposit, using a method commonly used in geology and whose application to tin deposits had been proposed earlier by Molofsky *et al.* (2014) by the use of isochrons (Albarède *et al.* 2012; Killick *et al.* 2020). The application of LIA to tin requires geological and geochemical modeling because in analyzing the tin ores (cassiterite) the *common lead* hypothesis is not valid (Sinha and Tilton 1973; Faure and Mensing 2005). This is rooted in the high content of uranium able to enter in the crystal lattice of the tin oxide, which is far higher than the amount incorporated in the sulphides normally forming the main ores of the metal deposits. Highly variable uranium contents may also result in highly heterogeneous distribution of radiogenic elements within cassiterite minerals from the same deposit (Swart and Moore 1982). The concept, long known in geochemistry, has been successfully applied by Molofsky *et al.* (2014) to South African, Botswanan, and Romanian artifacts. The method now has been applied to tin ingots from the Mediterranean area (Berger *et al.* 2019b).

The so-called “ancient tin problem” concerns the nature and origin of tin in Bronze Age Europe and the trade routes employed in the Mediterranean to supply tin-bronze production (Dayton 1971; Muhly 1985; Penhallurick 1986; Giunlia-Mair and Lo Schiavo 2003). The problem extends far eastward (Crawford 1974; Weeks 1999; Pigott 2011). The focus here is not to

review in detail the nature of the debate and the plethora of investigations attempting to solve the problem. Here we would like to remind a couple of methodological issues and appropriately review the existing LI data on relevant cassiterite deposits and Bronze Age tin objects.

Concerning methodology, the provenance of tin has long been investigated by the use of elemental tracers (Tylecote *et al.* 1989; Rapp *et al.* 1999) and tin isotope ratios (Gale 1997; Nowell *et al.* 2002; Hausteine *et al.* 2010; Yamazaki *et al.* 2014; Brüggmann *et al.* 2017; Nessel *et al.* 2019). Concerning chemical tracers, several elements (such as W, Bi, In, Mo) have proven potentially interesting to characterize cassiterite ore deposits. However, it has become apparent that there is yet insufficient chemical systematics of the relevant deposits and no adequate understanding of their behaviour during ore smelting. On the other hand, the variation in tin isotopes within a single ore district is comparable or larger than the difference between distant deposits. Any discrimination is therefore extremely difficult to obtain (Brüggmann *et al.* 2017). Isotope fractionation depending on reduction conditions may also be an issue (Berger *et al.* 2019a). As a matter of fact “the pioneering studies on tin isotopes carried out on some tin ingots from Hishuley Carmel, Kfar Samir south, Haifa and Uluburun have already revealed similarities and differences in the isotope composition [see references cited], but no conclusions could be drawn on the origin of the tin in those studies because of the lack of ore data.” (Berger *et al.* 2019a).

LI are then a promising method to solve the old question. Here we will briefly summarize the available LI data on cassiterite ores (Figs. 9,10 and Tabs. 2,3), tin ingots from the Eastern Mediterranean (Fig. 11), and some new data on tin ingots from the Central Mediterranean area (Fig. 12).

Tin ores

Fig. 9 shows the available data for cassiterite ores formed during the Variscan/Hercynian orogeny, that is the Late Palaeozoic collision between the Euramerican plate (Laurussia) and Gondwana. The data mostly comply with the model age of the event, dated between 360 and 280 Ma (see for example Shaw and Johnston 2016). A consistent date of about 320 Ma has been calculated by Berger *et al.* (2019a) and the isochron line reasonably fits most of the ore samples related to the Cornish, West Iberian, and Erzgebirge deposits. The LI data available for some tin ingots and metal scraps found in South West England (OXALID, also reported by Galili *et al.* 2013), and plausibly related to the Cornwall deposits, also show a reasonable fit to the Variscan model age (Fig. 9). However, several Iberian and Cornish cassiterite samples deviate from the reference isochron line, showing anomalously high values in the $^{207}\text{Pb}/^{204}\text{Pb}$ and/or in the $^{208}\text{Pb}/^{204}\text{Pb}$ ratios. They possibly indicate later events remobilizing the original mineralization or locally different geological sources of the metals included in the original mineral, characterised by different initial U/Pb and Th/Pb ratios. Clearly, full understanding of these anomalies would require ad hoc investigation and a more complex modeling with respect to a simple one-stage evolution of the Pb isotopes.

The LI data of the cassiterite ores from Sardinia (Villacidro and S. Vittoria: Tab. 3 and Fig. 9) are also rather interesting, because they are co-eval with the emplacement of the Arbus batholith (Zuffardi 1958; Biste 1982) and thus related to the Variscan event (appx. 290–320 Ma: Cuccuru *et al.* 2016). However, they seem to show a different trend with respect to the model 320-Ma LI curve, again indicating that a more complex geological model is needed. Furthermore, the LI data of most Sardinian cassiterite samples are very close to the values measured on the geologically related copper ores (Fig. 10). The same is true for the other tin-bearing deposits cited in the literature as possible sources of prehistoric tin (see for example Muhly 1985), that is, the cassiterite

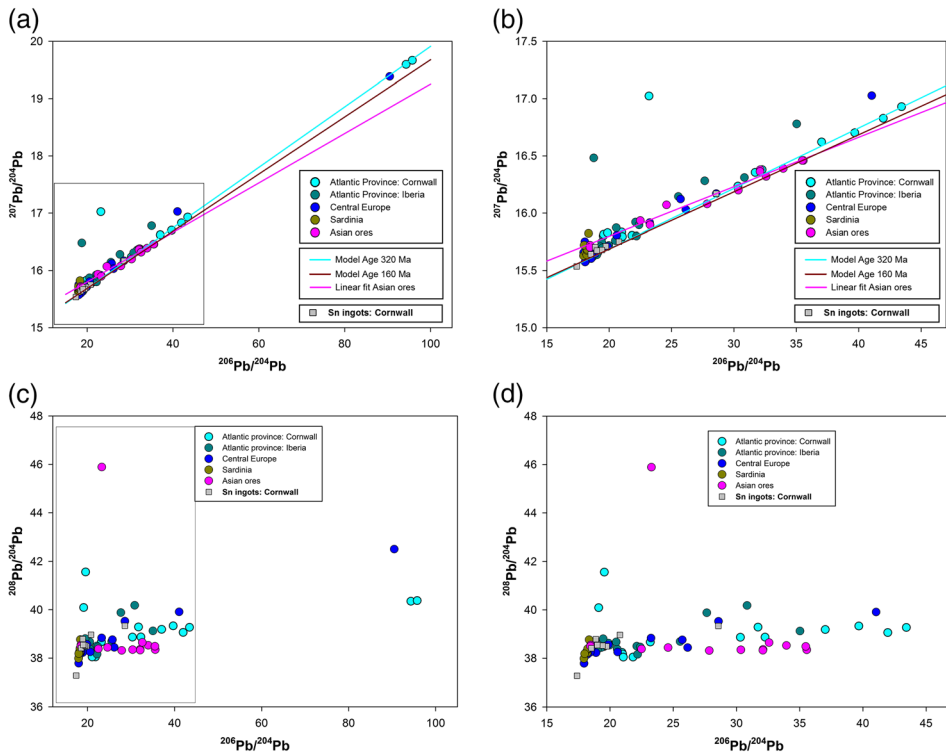


Figure 9 Available LI data for cassiterite ores. Figs. 9b and 9d are enlargements of the framed areas of Figs. 9a and 9c, respectively. Plotted LI data measured on ores related to the Variscan/Hercynian orogeny are Cornwall and Devon (OXALID), Iberia (OXALID, Marcoux 1997, Relvas et al. 2001, and our own measurements reported in Tab. 2), and Central Europe (OXALID, Niederschlag et al. 2003). The plotted data measured on Sardinian cassiterites from the Arbus batholith (Villacidro and S. Vittoria) are listed in Table 3. The plotted Asian ores are from China, Afghanistan, Kazakhstan, and Tajikistan (OXALID, Yuan et al. 2011), the linear fit through the Asian cassiterite data is shown as a magenta solid line. The isochron lines corresponding to deposits of 320 Ma (cyan line) and 160 Ma (dark red line) calculated by Berger et al. (2019a) and Yuan et al. (2011) respectively are reported in Figs 9a and 9b for reference. The data for tin finds (ingots) from Cornwall and Devon (OXALID, also reported by Galili et al. 2013) are also reported for comparison. [Colour figure can be viewed at wileyonlinelibrary.com]

ores in Tuscany (Monte Valerio: Stella 1955, Benvenuti et al. 2003) and in Anatolia (Kestel and Çamardi: Yener et al. 1989, but see discussion in Pernicka et al. 1992). This indicates that either time was too short or the contents of U and Th in cassiterite were too low to produce significant radiogenic Pb after cassiterite formation. The first hypothesis may apply to the 'young' Tuscan and Anatolian cassiterites, which are of Miocene (Poli 2004, Dini et al. 2002) and Late Cretaceous to Early Tertiary age (Alpaslan et al. 2006; Lermi et al. 2016), respectively. The second hypothesis is the most likely for the Sardinian cassiterite vein deposits. No matter what the geological cause, the lack of highly radiogenic samples precludes the use of the model age method to univocally discriminate these three deposits. Nonetheless, it should be noted that all three deposits show limited occurrences of tin ores, and there is to date no firm evidence of exploitation of any of them in prehistoric times.

The LI fields of Asian cassiterite deposits, taken into account as possible Eastern sources of tin, are mostly related to the Late Mesozoic Alpine-Himalayan orogeny. The linear trend

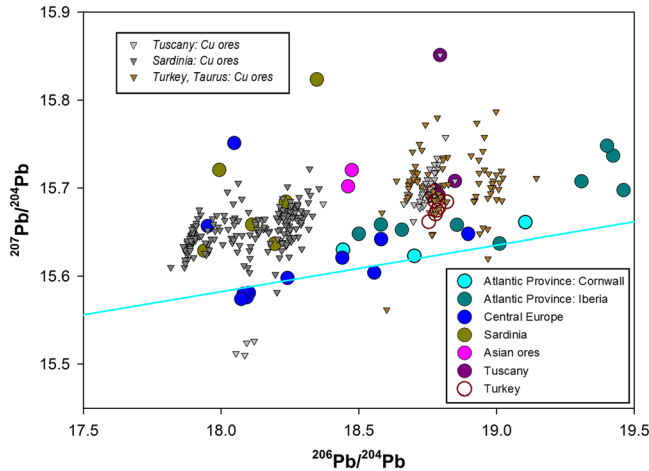


Figure 10 LI data for cassiterite ores (circles) from Sardinia (Villacidro and S. Vittoria, Table 3), Tuscany (Monte Valerio, Table 3), and Anatolia (Kestel and Çamardı, data from Yener *et al.* 1991). The isochron line corresponding to the 320 Ma model age of Variscan related deposits, as in Fig. 9. The LI data for cassiterites are compared to the LI data measured on copper ores from the same area, see text for details. [Colour figure can be viewed at wileyonlinelibrary.com]

Table 2 New LI data on Iberian cassiterites. Analytical error (2σ) refers to the last significant digit

Sample	Cassiterite ore	Reference	$^{206}\text{Pb}/^{204}\text{Pb}$	$^{207}\text{Pb}/^{204}\text{Pb}$	$^{208}\text{Pb}/^{204}\text{Pb}$
CS-1	Viana do Bolo, Ourense	Our measurement	20.492 ± 2	15.761 ± 1	38.675 ± 3
CS-2	Zona el trasquilo, Caceres	Our measurement	18.580 ± 8	15.658 ± 7	38.435 ± 17
CS-3	Hoyo de Manzanares, Madrid	Our measurement	19.461 ± 2	15.698 ± 1	38.808 ± 3
CS-4	Boiro, La Coruna	Our measurement	18.856 ± 2	15.658 ± 1	38.675 ± 3
CS-5	Logrosán Caceres	Our measurement	18.656 ± 3	15.653 ± 3	38.504 ± 7

Table 3 Available LI data for Sardinian and Tuscan cassiterite ores

Sample	Cassiterite ore	Reference	$^{206}\text{Pb}/^{204}\text{Pb}$	$^{207}\text{Pb}/^{204}\text{Pb}$	$^{208}\text{Pb}/^{204}\text{Pb}$
cass S	Villacidro	Our measurement	17.939 ± 2	15.629 ± 1	37.988 ± 3
sard 101	Villacidro	Begemann <i>et al.</i> 2001	18.113	15.658	38.249
MM 1004A	Villacidro	OXALID	18.195	15.637	38.227
MM 1004B	Villacidro	OXALID	18.232	15.676	38.362
MM 1004F	Villacidro	OXALID	18.235	15.684	38.396
	S. Vittoria	Ingo <i>et al.</i> 1998	17.993	15.721	38.186
	Villacidro	Ingo <i>et al.</i> 1998	18.348	15.823	38.768
Cass T	M. Valerio	Our measurement	18.775 ± 3	15.698 ± 1	38.965 ± 3
DUXS802	M. Valerio	Chiarantini 2005	18.849	15.708	39.012
C15064col	M. Valerio	Chiarantini 2005	18.795	15.851	39.266
99-CAS	M. Valerio	Chiarantini 2005	18.790	15.692	38.973

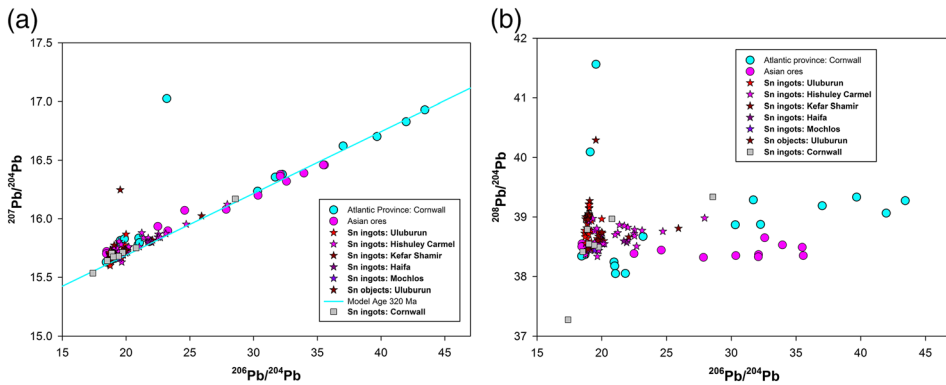


Figure 11 LI data measured on tin ingots (stars) recovered from shipwrecks in the Eastern Mediterranean: Uluburun, Hishuley Carmel, Kefar Shamir, Haifa, and Mochlos (Stos-Gale *et al.* 1998, Begemann *et al.* 1999, Galili *et al.* 2013, Berger *et al.* 2019a), compared with the ores (circles) and Cornish ingots (squares) also plotted in Fig. 9. The Iberian and Central European ores were omitted for clarity. [Colour figure can be viewed at [wileyonlinelibrary.com](#)]

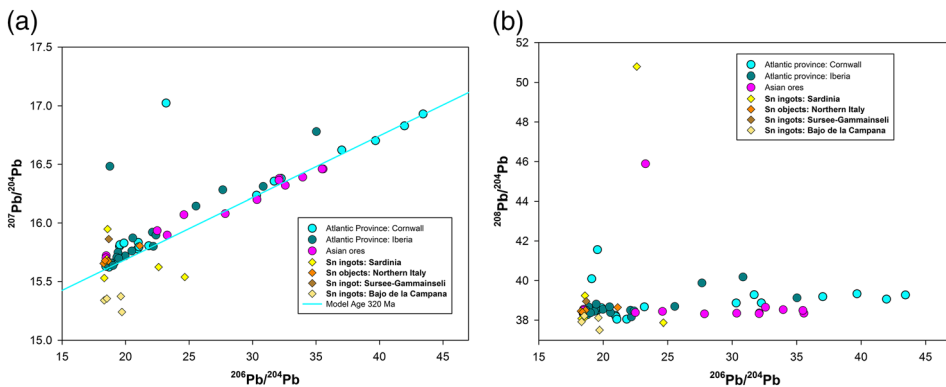


Figure 12 LI data measured on tin ingots and objects (diamonds) from Central Mediterranean compared with the ores (circles) plotted in Fig. 9. Objects are from Sardinia (Domu de S'Orku ingot: Ingo *et al.* 1998; tin ingots from S'Arcu, Table 4), Alba, Piemonte (tin wire, tin droplets: Angelini *et al.* 2018), Terramara di Parma, Emilia Romagna (tin bar ingot: Cremaschi *et al.* 2018), Sursee-Gammainseli, Luzern (tin ingot: Nielsen 2014), Bajo de la Campana (tin ingots: Mederos Martín *et al.* 2017). [Colour figure can be viewed at [wileyonlinelibrary.com](#)]

appearing in the plot (Fig. 9) is dominated by Chinese deposits, which are age modelled to about 160 Ma (Yuan *et al.* 2011; see dark red curve in Figs. 9a,9b). The two model ages of 320 and 160 Ma are very close at low $^{206}\text{Pb}/^{204}\text{Pb}$ and $^{207}\text{Pb}/^{204}\text{Pb}$ values, so they can hardly be discriminating for cassiterite provenance (Figs. 9a,9b). However, the overall slope of the linear fit to all Asian data (Figs. 9a,9b) is somewhat smaller than the 320 and 160 Ma model ages. This may be consistent with the younger age of the Asian deposits related to the Alpides phase of the Alpine-Himalayan orogenesis, which is Late Mesozoic to Cenozoic in age (Şengör 1986; Roland 2002). In any case, at this stage the substantial overlap between the Asian and Western European cassiterite LI data sets does not allow to use this difference as a discriminative tool. However, albeit the LI data available are scarce, it seems that there might be a chance of discriminating the Asian cassiterites with respect to the Variscan related deposits of Western Europe by using the $^{208}\text{Pb}/^{204}\text{Pb}$ vs $^{206}\text{Pb}/^{204}\text{Pb}$ plot (Figs. 9c,9d). In fact the data measured on cassiterite

samples from China, Kazakhstan, and Tajikistan (OXALID, Yuan *et al.* 2011) follow an almost horizontal line, possibly indicating a remarkably small amount of Th in the system and the subsequent negligible evolution of the $^{208}\text{Pb}/^{204}\text{Pb}$ ratio with time. It is unfortunate that no published LI data are available for the Iranian tin deposits, which have been repeatedly suggested as likely source candidates based on archaeological evidence (Nezafati *et al.* 2006; Cuenod *et al.* 2015). It should be noted that the only available datum for cassiterite from the Western region of Afghanistan (OXALID, Fig. 9) deviates from the apparent trend of the other Asian deposits and shows a very high value in the $^{208}\text{Pb}/^{204}\text{Pb}$ ratio (but not in the $^{207}\text{Pb}/^{204}\text{Pb}$ ratio), even much higher than the European Variscan deposits. Potentially, there are thus chances to discriminate Afghani deposits from other Asian sources, and this may be in line with the observations of Weeks (1999) of highly radiogenic tin in bronze objects from Tell Abraq, in the Arab Emirates region.

Tin objects

Inserting the numerous LI measurements of Sn ingots recovered from shipwrecks in the Eastern Mediterranean (Fig. 11) in the diagrams relative to cassiterite ores yields a number of information. (1) At least two sources of tin are present in the ingots, as well noted by previous authors (Galili *et al.* 2013, Berger *et al.* 2019a). (2) The great majority of the circulating tin is actually derived from Atlantic sources. In fact, even if in the $^{207}\text{Pb}/^{204}\text{Pb}$ vs $^{206}\text{Pb}/^{204}\text{Pb}$ diagram (Fig. 11a) the ingot data ambiguously show a reasonable fit with both the Cornwall and the Asian ores, in the $^{208}\text{Pb}/^{204}\text{Pb}$ versus $^{206}\text{Pb}/^{204}\text{Pb}$ diagram (Fig. 11b) most of them have a generally better agreement with the evolution line of the Western European ores and Cornish ingots. The conclusion agrees with those derived by tin isotope investigations (Berger *et al.* 2019a). However, part of the Uluburun and several of the Hishuley Carmel tin ingots cluster in a region deviating from the 320 Ma model trend, which is typical for most of the Cornish ores (Fig. 11a). This is also evident in the $^{208}\text{Pb}/^{204}\text{Pb}$ vs $^{206}\text{Pb}/^{204}\text{Pb}$ diagram (Fig. 11b), where some of the Uluburun ingots and objects display highly radiogenic character similar to some of the Cornwall cassiterites. This may indicate derivation from some anomalous Cornish ores, though this may also well indicate that some other mines in the Atlantic province (including Iberia) or in Variscan ore districts of continental Europe (such as the Massif Central or the Erzgebirge) were active in the Late Bronze Age. Moreover, although at present the LI evidence points to Western sources of tin for the Eastern Mediterranean ingots, we could actually speculate that the anomalous radiogenic signal observed for the Afghan deposits (discussed above) could even hint the possibility of a significant contribution of the Afghan tin to the composition of the Uluburun ingots.

Further, if we try to apply the developed LI method to the measured tin objects and ingots from the Central Mediterranean (Fig. 12), we obtain a similar picture as the one resulting from Eastern Mediterranean wrecked ingots. The LI data match rather well the fields defined by the Eastern Mediterranean ingots discussed above, so the same tin sources may be suggested, although the data are more scattered, indicating a variety of possible Western tin sources. Specifically: (1) the objects from Northern Italy (the tin wire and the droplets from Alba, Angelini *et al.* 2018; and the tin ingot from the Terramara di Parma, Cremaschi *et al.* 2018) fit rather well with the main trend of the Cornwall or Iberian ores. (2) The Sardinian ingots from Domu de S'Orku (Ingo *et al.* 1998), one of the ingots from S'Arcu 'e is Forros (SARC-43, Table 4), and the tin ingot from Sursee-Gammainseli (Nielsen 2014) seem to be related to Variscan sources characterised by variable Th/Pb and U/Pb ratios, thus departing from the 320-Ma model trend. Finally: (3) the remaining Sardinian ingots from S'Arcu 'e is Forros (SARC-42, SARC-

Table 4 Available LI data on Sardinian ingots. Analytical error (2σ) refers to the last significant digit

Sample label	Object	Reference	$^{206}\text{Pb}/^{204}\text{Pb}$	$^{207}\text{Pb}/^{204}\text{Pb}$	$^{208}\text{Pb}/^{204}\text{Pb}$
Panella Sn	Sn ingot, Domu de S'Orku	Ingo <i>et al.</i> 1998	18.5807	15.9482	39.2432
Sarc-42	Sn ingot, S'Arcu 'e is Forros	Pernicka, reported in Valera <i>et al.</i> 2005	18.5195	15.6920	38.1011
Sarc-42	Sn ingot, S'Arcu 'e is Forros	Our measurement	18.32 ± 3	15.53 ± 3	38.08 ± 6
Sarc-43	Sn ingot, S'Arcu 'e is Forros	Our measurement	22.6 ± 1	15.62 ± 8	50.8 ± 3
Sarc-46	Sn ingot, S'Arcu 'e is Forros	Our measurement	24.7 ± 2	15.54 ± 9	37.9 ± 2

46, Table 4) and the ingots from Bajo de la Campana (Mederos Martín *et al.* 2017) have rather low $^{207}\text{Pb}/^{204}\text{Pb}$ and $^{208}\text{Pb}/^{204}\text{Pb}$ values, possibly indicating a different, relatively unradiogenic source.

Concerning the diffusion of tin in the Mediterranean, the generally accepted narrative of tin metallurgy rising in Anatolia in the early part of the third millennium BC and linearly expanding westward at the end of third millennium BC does not encounter solid confirmation, based on LI data. Well before the massive spread of tin-based alloys in the Bronze Age, the existence of tin metallurgy in the 5th millennium BC has been discussed in the frame of the evidence of polymetallic metallurgy related to the Vinča culture of the Balkans (Radivojević *et al.* 2013). There are thus challenges to the conventional model of Eurasian metallurgy. Moreover, the increased content of tin in the blades of the younger graves of the Singen cemetery (Early Bronze Age) seems to be related to the appearance of Armorico-British daggers, hinting to an Atlantic source of tin (Krause 1989). The above discussion of the available LI data, supporting of the presence of Atlantic tin in the recovered ingots from the Turkish and Israeli coasts reinforces the model of tin flowing from the mythological “Cassiterides” islands located in the West during the Bronze Age. Expanding work on the Iberian and Balkan ore deposits may well show that there might even have been multiple sources of the metal (Huska *et al.* 2014; Comendador Rey *et al.* 2017), further increasing the complexity of the scenario for the metallurgical production and diffusion. The tin ores need many more LI measurements in order better investigate their inhomogeneity and practically define reference “fields” and “trends” that can effectively be used for provenancing studies.

CONCLUDING REMARKS

LI data are nowadays an important part of metal provenancing investigations. The application of isotopic tracers has expanded to interpret metal production, diffusion, and trade at different scales, from the local level to the regional or to long-distance movements. In combination with chemical tracers and other developing isotopic systems (Sn, W, Fe, Sb, Os, Nd, etc.) LIA provide a powerful tool to explore the past movement of materials, technology, ideas, and, of course, people. The selected case studies discussed in this paper support the idea that isotopes

are increasingly helping to define and quantify the details of the metal flow in space and time, and this in turn enhances our understanding of human thinking and mobility (Martinon-Torres in Armada *et al.* 2018). The net result is that the picture of ancient metal production and trade is getting less blurred, more detailed, and far more dynamic and complex than perceived in the past.

However, the consequence is that in practice we need to deal with an increasing amount of technically and instrumentally sophisticated data, and often the conceptual and technical interpretive tools are not adequate to the needs. In the specific realm of archaeometallurgical data, at present we are greatly suffering from the limitations posed by databases and methods of inquiry. The existing data sets are incomplete, biased, and poorly interconnected. Unless there is some critical innovation in the management and integration of chemical, isotopic, and archaeological data, soon we'll not be able to efficiently relate and interpret the data that are increasingly available. We will measure data that we might not be able to adequately compare with existing information. It should be a priority to standardize, rationalize, and make publicly accessible the huge amount of existing data already available. Journals should require data deposition in standardized form and much more effort should be put into software for quality assessment, data consistency, and efficient interrogation. This is being actively pursued in other fields, but it is hard to come by in archaeology and archaeometry for a variety of historical and specific reasons, not least the critical mass of research groups. The small step that the Isotrace Laboratory in Oxford did when making OXALID publicly accessible had enormous consequences for the diffusion of LI methods to research groups not able to develop internal databases. If we want to move forward, it is now time for all archaeometallurgists to share information and enter in the regime of big data analysis.

ACKNOWLEDGEMENTS

Igor Villa and Kris Latruwe are acknowledged for continuous support and collaboration with LI measurements in Bern and Ghent, respectively. Giancarlo Cavazzini is warmly thanked for continuous support on data preparation and clean-room maintenance. Ignacio Montero provided samples of Iberian cassiterites and Sardinian tin objects from S'Arcu 'e is Forros. Marica Venturino is acknowledged for the exciting collaboration on the objects from Alba. Zofia Stos-Gale kindly made most of the OXALID data available for data analysis. Piero Guzzo, Danilo Franco, Fabio Demasi, Bruno Furina, Luigi Dattola and Sara Marino kindly provided ore samples from Longobucco. Ignacio Montero and Mark Pearce contributed a number of useful suggestions that greatly improved the early draft of the paper.

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