# LEAD ISOTOPE ANALYSIS AND CHEMICAL CHARACTERIZATION OF METALLIC RESIDUES OF AN EARLY BRONZE AGE CRUCIBLE FROM GÖLTEPE : USING ICP-MS

## KURŞUN İZOTOP ANALİZLERİ VE GÖLTEPE İLK TUNÇ ÇAĞ'I POTASINDAKİ MADEN ARTIKLARININ KİMYASAL NİTELİĞİ: ICP- MS KULLANIMI

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**Key words:** Lead isotope, crucible,provenance, Early Bronze Age, Göltepe, Anatolia
**Anahtar Sözcükler:** Kurşun izotopu, pota, kaynak araştırması, İlk Tunç Çağ, Göltepe, Anadolu

## ÖZET

*Bu çalışmanın öncelikli amacı, Göltepe'deki bir İlk Tunç Çağ pota parçasında bulunan metal kalıntılarının kurşun izotop oranlarını bilinen pek çok metal madeninden edinilmiş verilerle karşılaştırarak pota kalıntısındaki kurşunun hangi kaynaktan gelmiş olabileceğini saptamaktır. Pota parçası üzerinde yapılan kurşun izotop ölçümü, metal kalıntılarının uzak maden yataklarından mı, yoksa, varsayıldığı gibi, Toros Dağları'nda bulunan daha yakın kaynaklardan mı geldiğini belirleyebilmek için eşsiz bir imkan sunmaktadır. Elde edilen ölçümler, bilinen Toros madenleriyle kısıtlı bir ilişkinin varlığına işaret etmektedir. Buna ek olarak, metal kalıntıların ppb'sindeki elementleri incelemek amacıyla, element kompozisyon analizleri yapılmıştır. Araştırma sonuçları, Anadolu'nun bu yöresinde İlk Tunç Çağ boyunca görülen, madencilik ile ilgili iş bölümü ve organizasyonuyla uyumluluk göstermektedir.*

## INTRODUCTION

Changes in the organization of metallurgical technologies of the third millennium BC in Anatolia appear to coincide with overarching socioeconomic and political changes in urban regions that surround or are adjacent to highland resource areas (Yener 2000). Early local highland developments in metal technologies, however, likely include indigenous cascades of innovation and long-term practical familiarities with metal producing regions. Network-based relations among these areas, including lowland urban agglomerations, farming communities and highland mining regions, were likely structured in part on access to valuable metal resources. The structure of these relations seem to vary over time, where, by the third millennium BC, economically specialized communities lived within highland mining regions to participate in the extraction, preparation and smelting of locally available metal resources. The site of Göltepe, an Early Bronze

Age settlement situated in the Central Taurus region of southern Turkey ca. 2600 BC, represents the first tier of the multi-scale production of metal. A primary industrial activity at the site was the smelting of tin ore, which is evident from ore crushing tools, processed tin-rich powders, hundreds of excavated crucibles with tin accretions (Yener and Vandiver 1993; Earl and Özbal 1996; Yener *et al*. 2003). In addition, the presence of locally available tin from the Kestel mine, roughly two km northwest of Göltepe, suggests that inhabitants at the site must have been familiar and utilized these resources.

Chemical analyses of major and minor elements of the iron-rich powders and hematites with tin by Earl and Özbal (1996: 296) do suggest similar sources based on their similar weight percentages. However, lead isotope analysis provides an additional line of evidence for discerning this potential similarity. This paper focuses on the later assumption, testing it by use of lead isotope analysis of the tin-rich accretions of one crucible. Our goal is to compare the isotopic fingerprint provided by the analysis of the crucible residues to known signatures of previously analyzed ore bodies in Anatolia. We conclude that the isotopic signatures of the metalliferous residue are compatible with the Central Taurus ore bodies and thus provide evidence of local acquisition of materials.

## CONTEXT: 'EARLY BRONZE AGE MINERS' VILLAGE GÖLTEPE

The third millennium BC miners' village, Göltepe, was excavated during 1990, 1991 and 1993 as part of a broader archaeometallurgical research project in the central Taurus Mountains (Yener 2008, in prep). In the immediate environs of the site, the Kuruçay stream emerges from the Niğde massif to the north, carrying with it cassiterite, pyrite, hematite, garnet, tourmaline, magnetite, sheelite, cinnabar, titanite, rutile, apatite, monozite and gold. The Niğde Massif, a volcanic dome within the Taurus Mountain chain, is to the SE of the vast Niğde plains (Fig. 1). The region is an area of intense plate tectonic activity with a succession of subduction zones and consequently mineralization is common in the area. The third millennium BC Kestel tin mine is located on a slope which is pervaded with granite, marble, gneiss and quartzite. The tin ore has been worked in marble which appears to have been originally part of a Triassic sequence of dolomitic limestone. Iron in the form of hematite, magnetite and limonite is visible in the mine and the cassiterite ores were derived from a granite intrusion process.

Göltepe hill is located at 1767 meters above sea level, measures ca. 60 hectares total and is fortified at the summit (Fig. 2a). This well protected miner's village is located two kilometers opposite Kestel tin mine and the size of the intramural settlement are estimated to be between 8-10 hectares. Occupied from Early Bronze I-III, the pit house complexes and rock cut terraced architecture typified a cottage industry processing tin ore from Kestel mine as well as other ores from the polymetallic sources of the region. This production was a labor intensive process of grinding the iron-rich cassiterite into powder consistency before smelting was achieved in crucibles or bowl furnaces with blow-pipes and tuyères. The product, a rough mix of tin metal prills and iron was then enriched by washing, repeated grinding and resmelting.

Crucible M325 (context number B01-0107-003), stems from slope wash abutting the base of a well-built N-S circuit wall (B01-0100-004), which was preserved in some places to ten courses and a height of over a meter (Fig. 2b and 2c). This context yielded hundreds of variously sized tin encrusted crucible fragments weighing over 300 kg.

## LEAD ISOTOPE ANALYSIS

The sensitivity of lead isotope ratios coupled with the fact that measureable amounts of lead occur in many materials, including most if not all archaeological metals, allows for a robust method to study material alteration, production, transport and provenience if conducted carefully (Weeks 2004: 129-133; Aggarwal 2008: 2662; Villa 2009). The geological history of Anatolia (see Öztunalı 1989; Okay 2008) is punctuated by discrete events in which the conditions for ore formation processes were established. Particular mining regions (e.g., the Bolkardağ, see Yener *et al*. 1991) where metal bearing ores are most abundant and concentrated may then have specific isotopic fingerprints that relate to their geological histories. However, given that similar

ore formation processes existed across many regions of Turkey, it is expected that these regions will also have similar lead isotope fingerprints.

The element lead (Pb) has four stable isotopes $^{208}Pb$, $^{207}Pb$, $^{206}Pb$, and $^{204}Pb$. Of these four isotopes, only $^{204}Pb$ is non-radiogenic where $^{208}Pb$, $^{207}Pb$ and $^{206}Pb$ are formed by the regular decay of $^{232}Th$, $^{235}U$ and $^{238}U$ respectively (Faure and Mensing 2005: 214-249; Allègre 2008: 294-312; Tolstikhin and Kramers 2008: 385-390). The relationship among these four isotopes of lead, which are often expressed in ratios, theoretically allow for the calculation of geological age (not archaeological age) based on principles of radiometric geochronology (e.g., U-Pb and Pb-Pb system dating techniques). There exist three important conditions concerning the study of lead isotopes to characterize sources and artifacts:

1.) The relative concentrations of these four isotopes are assumed to have been uniform throughout the earth at the time it was formed;
2.) Isotopes $^{208}Pb$, $^{207}Pb$, and $^{206}Pb$ have continued to be formed on earth from the radioactive decay of uranium and thorium. Therefore, naturally occurring lead is composed of a diagnostic mixture of radiogenic and original terrestrial lead
3.) Ratios of lead isotopes vary depending on the geological age of the ore body and the conditions under which it mineralizes, the proportions of which vary among geological formations (Guilbert and Park 2007: 286-290).

Additionally, because different ore sources often have unique lead isotope ratios according to a specific geological origin, *"variation among different sources can in some cases be greater than variation within sources"* (Weigand *et al*. 1977). Under these conditions, the isotopic analyses of archaeological metal and metal-rich residues, which often contain measurable amounts of lead, can be compared to lead isotopes from known ore sources (Brill and Wampler 1967; Gale and Stos-Gale 2000; Stos-Gale 2000). Strong similarities among artifacts and ore sources can be used as evidence for their provenience.

While much debate surrounds the nature of artifact-ore relationships and how one ascertains these relationships (e.g. Pernicka 1995; Budd *et al*. 1996; Knapp 2000; Gale 2001; Begemann and Schmitt-Strecker 2008;

Baron *et al*. in press) for the purposes of this study, I will judge artifact-ore relationships in terms of source discrimination. Since geographically distinct ore sources can have very similar lead isotope ratios, similarity in isotope ratios does not necessarily equate to congruity in artifact-ore relationships. For this reason, this study aims to eliminate as many conceivably utilized sources as possible based on comparative isotopic signatures of artifacts and ore bodies.

## METHODS

### SAMPLE PREPARATION

The crucible was prepared for acid dissolution by taking separate drillings that were separately digested in nitric acid. Six drillings were taken from the Göltepe crucible fragment with an electric tungsten steel hand drill. The drillings consisted of about 1 cm long extractions taken from areas of the crucible that appeared to have visible metal-rich areas (see Fig. 3). The crucible was drilled twice on the exterior (#5.1-5.2) as control samples and four times on the interior (#5.3-5.6), so that comparable measurements may be taken. 1 to 3mg of crucible drillings from each area were then placed into 2 ml vials to which was then added .25 mL of high concentration nitric acid. All the samples were allowed to digest over a period of three days to ensure a more complete digestion of the crucible drillings. After this period of time, all of the samples were centrifuged, and the remaining liquid was carefully removed using a sterile pipette and put into a new sample vial to which was added 4.8 mL of distilled water and shaken. The result was 5 ml of diluted solution. Although nitric acid does not dissolve silicate minerals and this digestion leaves considerable residue, enough lead was solubilized to obtain precise isotope ratio measurements.

### *ICP-MS PROCEDURES*

We analyzed lead isotopes of metalliferous residues using a Finnigan MAT Element I high-resolution magnetic sector ICP-MS at the Laboratory for Archaeological Chemistry, University of Wisconsin – Madison. Accuracy of the ICP-MS measurements is accomplished by comparison of known actual ratios to the average measured ratios of several assayed NIST standards (National Institute of Standards and Technology). A correction value, a

value generally slightly above or below the nominal standard, was then computed by subtracting the NIST assays from the nominal standard. These correction values would then be added accordingly to the artifact sample ratio values. Additionally, because each sample's ratio values are based on repeated measurements (n=50), a relative standard deviation (% RSD) was computed. The % RSD is a measurement of precision; a higher value equates to a sporadic and generally dispersed nature of the isotope counts. Low values, where % RSD is generally less than 0.5 %, equates to a relatively non-sporadic spread of the isotope counts, and is a more *precise* measurement. Measured ratios of NIST 981 wire standard compared to the known nominal ratios produced relative standard deviations less than 0.2 % (see table 1), which is appropriate for lead isotope analysis.

The crucible samples are assayed after the level of precision was established and the ICP-MS capillary tubing was flushed. An exploratory assessment of several elements potentially contained within the sample solution was completed. This exploratory analysis measured selected elemental compositions of the residues extractions, namely arsenic (As), copper (Cu), nickel (Ni), lead (Pb), tin (Sn), silver (Ag), niobium (Nb), and tantalum (Ta). Each sample was assayed once and was separated by a period of 30 seconds of downtime in order to prevent contamination. A pitfall of this procedure was that the digestion was not a total digestion and, hence, the results are not quantitative, but merely reveal any anomalous excess or deficit of these elements. Actual concentrations of the elements of the sample could not be determined. This exploratory analysis nonetheless provides an insight into the elemental make-up of the sample solution and thus an insight into the chemical make-up of the artifact sample, which is ultimately a potential reflection of the physical character of the metallic residues. The results are presented below in Table 2.

The four lead isotopes were measured using the same sample solution as the exploratory analysis and the ratios are listed in Table 3. Based on expectations, an effort was made to assay each sample according to the expected level of lead concentration; that is, samples thought to have less lead were run first. The results of the lead isotope analyses were then presented in a standard format

that allows for the comparison with the results of others. This format is in the form of isotope ratios. Because there are only four isotopes of lead, there can only be three distinct ratios among a dozen possibilities. While the specific isotope ratios chosen by individual authors may vary, the entirety of the ratios is easily extrapolated from presented forms by a means of common algebra. Those isotope ratios presented in this paper are as follows: $^{208}Pb/^{206}Pb$, $^{207}Pb/^{206}Pb$, and $^{204}Pb/^{206}Pb$. Additionally, only the ratios $^{208}Pb/^{206}Pb$ and $^{207}Pb/^{206}Pb$ will be considered in this paper because of the high % RSD associated with generally lower CPS values for the isotope $^{204}Pb$. The higher % RSD value for $^{204}Pb$ means that any ratio extrapolated from this value would be considerably less accurate.

## RESULTS AND DISCUSSION

The Göltepe samples had expectedly low counts of all metals given the diluted nature of the digestion (Table 2, Figure 4), however with generally higher amounts of nickel and tin for the interior drillings #5.3-5.6. It should also be noted that the exterior drillings #5.1-5.2 had up to three times as much lead as the interior, but only trace amounts of tin. The cause of this difference is probably a due to the sampling strategy employed and the heterogeneous character of the residues on the interior surface of the crucible. The Göltepe sample appears to correlate with the other crucible analyses (Yener 2000; Yener and Vandiver 1993; Yener *et al*. 2003), where significant amounts of tin and copper are present. The amounts of lead from the interior of the crucible provide ample quantities for accurate measurements of lead isotopes.

The results for the two ratios, $^{208}Pb/^{206}Pb$ and $^{207}Pb/^{206}Pb$, were plotted in a series of bivariate scatterplots in order to visually observe differences and possible correlations with previously published lead isotope data of known Anatolian ores (see Figs. 5 and 6). Ores are presented with 95% confidence ellipses drawn using the XLSTAT addin for Excel 2007. A multinational effort over the last two decades has accomplished many characterization studies that amounted in the present database of lead isotope ratios of known ore bodies in Turkey. Currently, over 300 ore samples have been published and have been used to characterize known bodies of metal ore (Brill and

Wampler 1967; Begemann *et al.* 2002; Stos-Gale and Gale 1981; Gale *et al.* 1985 Hirao *et al.* 1995; Kuovo 1976; Pernicka 1984; Sayre *et al.* 1992, 2001; Seeliger *et al.* 1985; Vavelidis 1985; Wagner *et al.* 1985, 1986, 1992, 2003; Yener *et al.* 1991). An expected degree of overlap in lead isotope ratio values certainly occurs among many of the sources, however there is a noticeable but not perfect separation of values between Central Anatolian, Pontide and Taurus based ore bodies.

Results indicate that that North Central Anatolian and Pontide ore sources are incompatible with the measured lead isotope ratios (see Fig. 5). Given the geographic location and nature of the social organization and technologically specialized settlement of Göltepe, Occam's Razor eliminates furthest sources when locally available sources are abundant. Much more likely would be the local exploitation of the rich metal resources in the surrounding Taurus Mountains. This is further suggestive with work of Yener and Vandiver (1993) and Earl and Özbal (1996) where similarities in major and minor elemental constituents between the Kestel mine and Göltepe production materials are compatible with similar sources. Similar elemental chemistry, proximity to nearby sources and the apparent specialization of ore processing and smelting at the site lead us to expect that the isotopic signatures should also be compatible with the Central Taurus ore bodies. The measurements are compatible with the Taurus groupings (see Fig. 6), specifically both Taurus 1A and 2B, however two measurements appear to be outliers. Compared to ore samples taken at Kestel (Yener *et al.* 1991: AON399, AON 419, AON463, and AON466), the crucible residue measurements overlap with AON 419 in the Taurus 2B ore group. The other three Kestel samples, however, are situated within the Taurus 1B grouping, which does not comprise a compatible group with the crucible residue measurements.

## CONCLUSION

In this paper, we have demonstrated the possibility of extracting elemental data from metalliferous residues from a crucible using an ICP-MS. Two major analyses were completed in order to better understand the metal composition of the residues. The first analysis consisted of the measurement in ppb of several elements that would help characterize the chemical nature of the crucible residues. The most prominent metals included tin and nickel. Trace levels of lead were also measured, which allowed us to measure isotope ratios of lead for provenance analysis. Because lead isotope ratios are a known indicator of possible provenience, we compared our ratio data with published lead isotope ratios. Our data indicate with independent chemical perspective that individuals and communities who once lived at the site of Göltepe actively acquired nearby raw and/or processed ores for use in the first tier of complex metallurgy in the region. Further, these data help substantiate upon the notion that Early Bronze Age cultures in Anatolia were forming economically specialized communities in highland metal resource regions that linked lowland urban centers by trade and exchange.

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| Ratio | Nominal | Measured | ± | %RSD |
|-------|---------|----------|-----|------|
| $^{208}Pb/^{206}Pb$ | 2.1681 | 2.1742 | 0.002 | 0.09 |
| $^{207}Pb/^{206}Pb$ | 0.9146 | 0.9163 | 0.001 | 0.11 |

Table 1: NIST 981 Lead Wire Standard. Note that relative standard deviations are less than 0.2 %.

| Sample # | Ni | Cu | As | Nb | Ag | Sn | Ta | Pb |
|----------|-----|-----|-----|----|----|------|----|----|
| #5.1 | 17225 | 708 | 83 | - | - | 64 | 3 | 19 |
| #5.2 | 6508 | 350 | 119 | - | - | 226 | 5 | 27 |
| #5.3 | 16312 | 228 | 11 | - | - | 420 | 2 | 4 |
| #5.4 | 10290 | 196 | 3 | - | - | 546 | 2 | 10 |
| #5.5 | 6835 | 233 | - | - | 2 | 3618 | - | 4 |
| #5.6 | 6845 | 301 | - | - | 3 | 4485 | - | 3 |

Table 2: Traces of several elements (in ppb) measured from metalliferous residues from the Göltepe crucible. Sample #5.1-5.2 was taken from the exterior of the crucible, while samples #5.3-5.6 were taken from the interior

| Sample # | Artifact ID | $^{208}Pb/^{206}Pb$ | $^{207}Pb/^{206}Pb$ |
|----------|-------------|---------------------|---------------------|
| #5.1 | MRN-325 | 2.0615 | 0.8281 |
| #5.2 | MRN-325 | 2.0520 | 0.8235 |
| #5.3 | MRN-325 | 2.0500 | 0.8241 |
| #5.4 | MRN-325 | 2.0425 | 0.8261 |
| #5.5 | MRN-325 | 2.0631 | 0.8370 |
| #5.6 | MRN-325 | 2.0630 | 0.8288 |

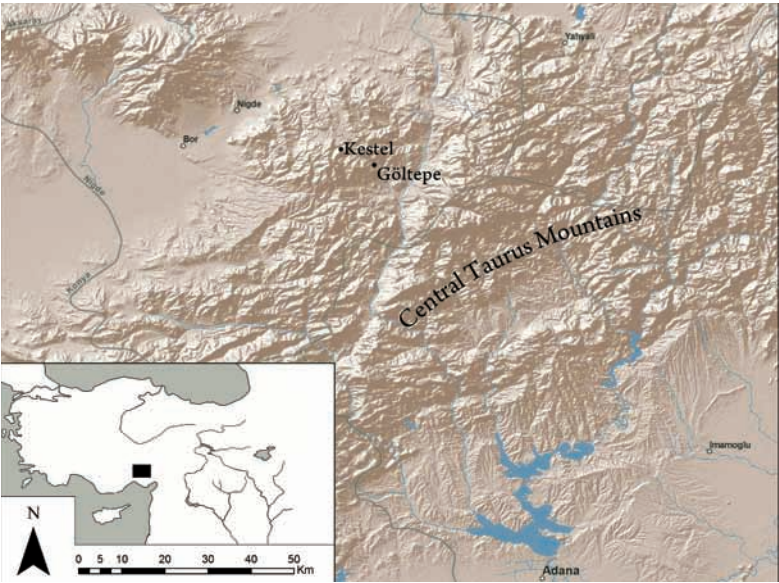Table 3: Lead isotope ratios measured from the interior and exterior of the Göltepe crucible.

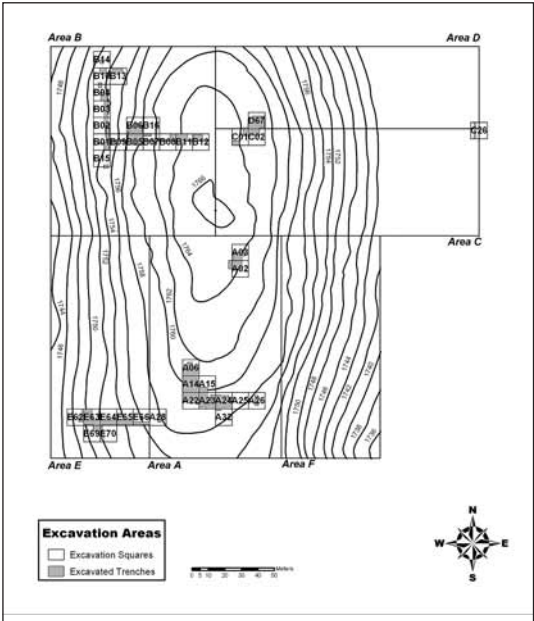Fig. 1: Geological specimen location map of the Central Taurus (Adapted from Yener et al. 1991:

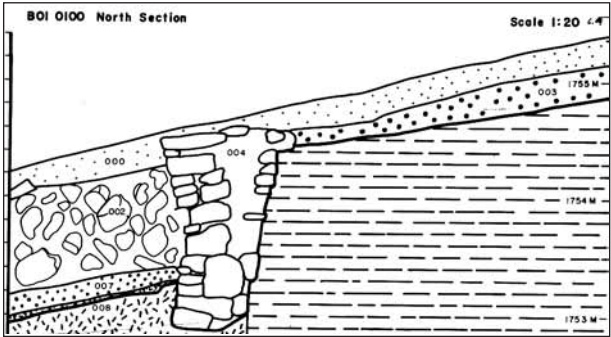

Fig. 2a: Excavation Areas Göltepe



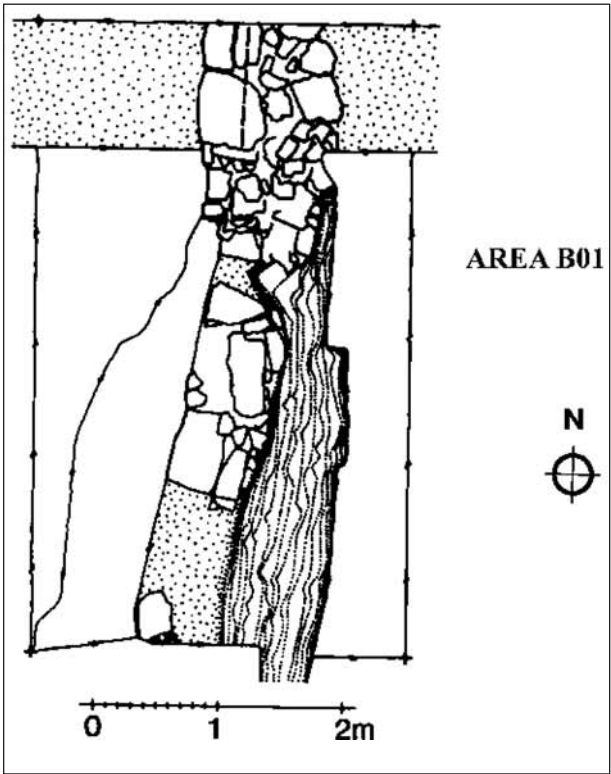Fig. 2b: Cross section of circuit wall. Crucible MRN 325 in locus B01-0100-007.



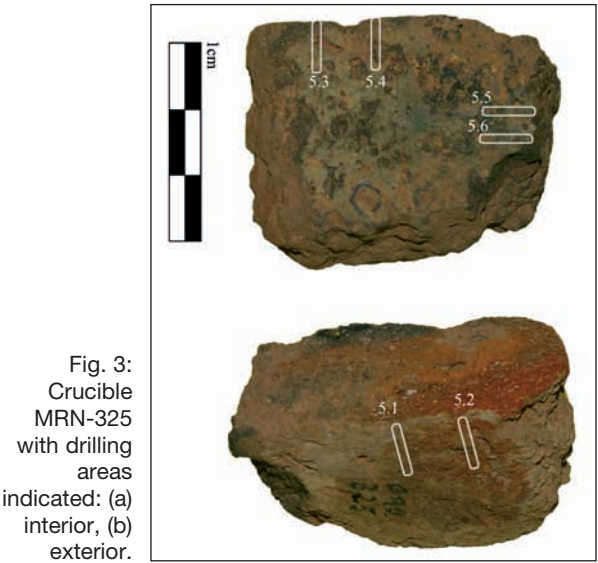Fig. 3: Crucible MRN-325 with drilling areas indicated: (a) interior, (b) exterior.
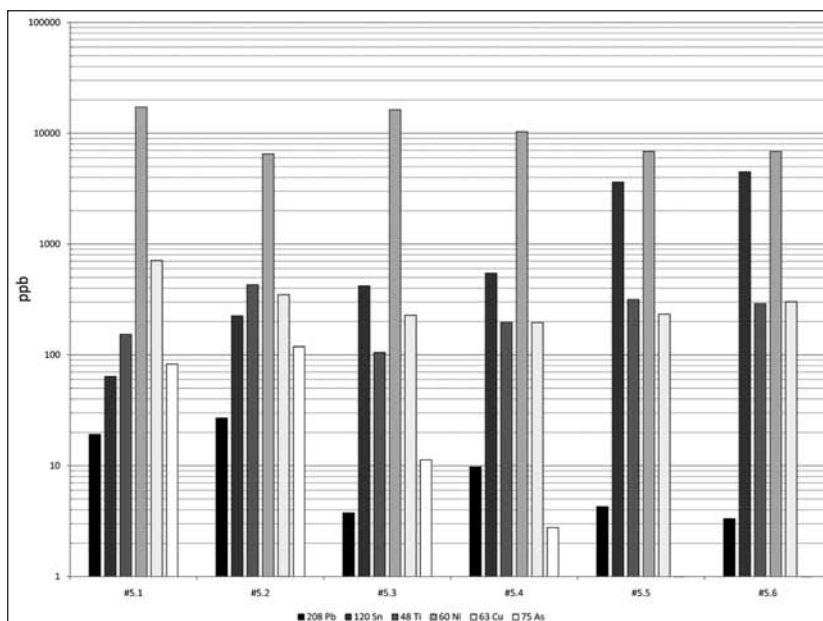


Fig. 2c: Circuit Wall Area B01

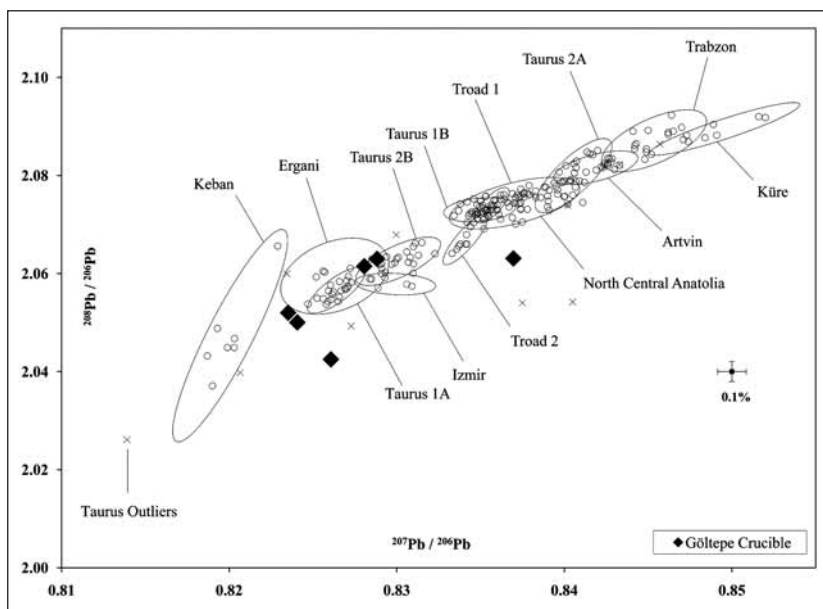Fig. 4: Elements measured in ppb from Crucible MRN-325.



Fig. 5: Scatter plot of lead isotopes in a larger context overlaid by lead isotopes measured from crucible MRN-325.
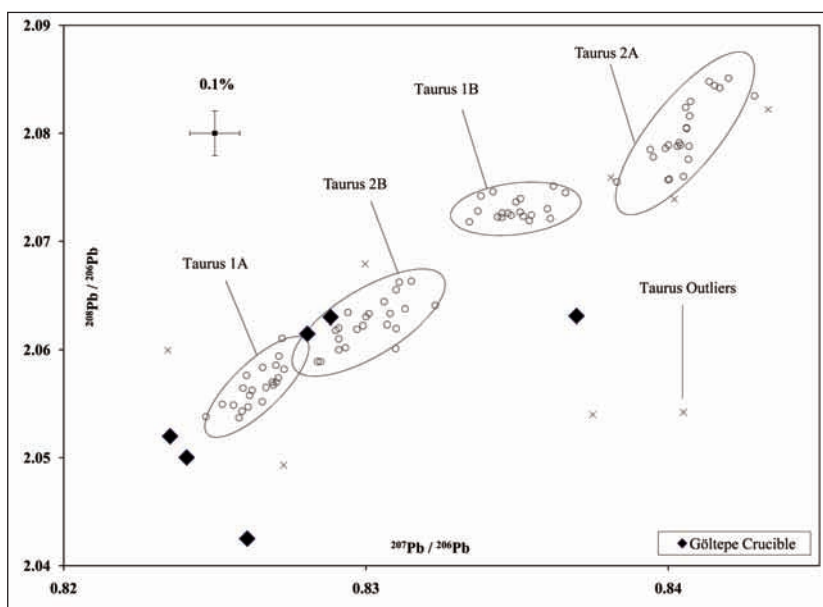


Fig. 6: Scatter plot of lead isotopes measured from Taurus ores overlaid by lead isotopes measured from crucible MRN-325.