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ICP Analyses from the Cinnabar-Mercury Occurrence at Azogues (Loma Guashon), Ecuador: Ancient Industrial Uses and Human Health Implications

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Abstract

ICP (Inductively Coupled Plasma) analyses of the Cretaceous marine sandstones at the Azogues (Loma Guashon), Ecuador cinnabar-mercury occurrence gave 11 - 113 ppm Hg. However, the ancient Azogues mines have been well documented previously, for example: 1) a 1799 hand drawn map "Plan del Cerro Mineral de *Azogue*" shows numerous adits; 2) during Colonial time, Cuenca founder, Gil Ramirez Davalos was owner of the mercury mines; 3) in the late 1800s, Teodoro Wolf described well-worked adits at Guashon and samples with 0.5% Hg; and more recently, 4) the Metallogenic Map of Ecuador shows the mercury occurrence at Azogues. Mineral resource assessment of the Azogues occurrence is important to regional archaeological studies of resource availability and ancient use of cinnabar as a pigment and as a source of mercury for gold amalgamation before the arrival of the Europeans, and possibly later, for silver amalgamation, during Colonial time.

Keywords

Gold, Mercury, Ecuador, Alluvial, Pre-Columbian

1. Introduction

Mercury and cinnabar occurrences are known throughout the Andes and blood-red cinnabar (HgS), the ore of mercury, was mined, selectively sorted, and ground for use as a pigment (vermilion), in funeral rituals, and as makeup during pre-contact time (Whitaker, 1941; Petersen, 1970/2010; Brown, 2001; Brooks et al., 2008).

Alluvial gold was ancient man's main source of gold (Boyle, 1979), and Ecuador's rivers and streams were the likely sources of Ecuador's pre-Columbian gold and platinum (Petersen, 1970/2010; Cárdenas, 1990; Valdez et al., 2005; Carrión Mero et al., 2011; Lleres Peréz, 2015). For example, the Longroño placers, southeast of Cuenca, at the headwaters of the Rio Santa Bárbara, were identified as an alluvial gold source and the Laguna de Ayllon may contain fine-grained gold flakes washed from native chiefs during ancient rites (Yepez, 2016).

Mercury was used to amalgamate the millimeter-sized, and smaller, gold flakes (*chispitas*) found in these alluvial occurrences (Holloway, 1932; Delbridge & Robertson, 1992; Báez, 2014) and elsewhere in the Andes before the arrival of the Europeans (Cabrera la Rosa, 1954; Kaufmann-Doig, 1978; Larco Hoyle, 2001; Brooks et al., 2013). Any gold nuggets, though rare, would have easily been removed from the gold pan (*batea*); however, the use of mercury is the only way to selectively remove the gold flakes from the ubiquitous "black sand" or heavy mineral concentrate (magnetite, cinnabar, zircon, rutile, and garnet) remaining in the gold pan. Small-scale gold mining, with mercury, of these fine-grained alluvial gold deposits continues to provide gold to Ecuador's modern mining economy (Gemuts, 1992; Sandoval, 2001; The Economist, 2013; Kiefer et al., 2015).

Regionally, Perú produced 16-22 metric tons of gold from small-scale alluvial gold mining, using mercury, during 2007-2011 (Gurmendi, 2012). And, as another example of the importance of mercury in alluvial gold mining, approximately 26,000,000 pounds (or 342,000 seventy-six pound flasks) of mercury were used during the California Gold Rush in 1849-50 (Alpers et al., 2005).

2. Azogue and Mercury

Azogue is a widely used synonym for mercury in the many small-scale alluvial gold mining areas throughout Latin America (Brooks, 2014). The word *azogue*, and variations, have been applied as toponyms, for example, *Azogues*, Ecuador, *Azogines* in Puno, Perú (Petersen, 1970/2010), and *Azogani* in Bolivia (Ahlfeld & Schneider-Scherbina, 1964). Both *azogue* and cinnabar (*zinjafir*) have roots in the Arabic occupation of Spain and *azogue* was the name used for the silvery liquid metal that was produced from the world's oldest mercury mine, Almadén (*al-Maden* or The Mine, also Arabic in origin), in Spain (Bennett, 1948). Even now, in Latin America, Spanish mercury is preferred for small-scale gold mining and *azogue*, with its hint of Spanish origin and supposed higher quality, commands a higher price than recycled local mercury or mercury produced in the U.S. or elsewhere.

3. Regional Mercury Occurrences

Petersen (1970/2010) indicates that there are least 20 mercury occurrences in Perú, one of which is Azogines in Puno; however, the most well-known and important occurrence in the region is the Santa Barbara mercury mine, Huancavelica, Perú (Arana, 1901; Yates et al., 1955; Brown, 2001); however, Huancavelica mercury production stopped in 1975 (Roskill, 1990). In Bolivia, there are three mercury occurrences that include: 1) Mina María Paz, near a hill known as *Azogani*; 2) Mina El Triunfo, near Oruru; and 3) Mina Emilia, near Palca de Higuera (Barba, 1640/1923; Ahlfeld & Schneider-Scherbina; 1964). There are numerous occurrences in Colombia (Singewald, 1950; Morer & Nicholls, 1962; Hall & Feininger, 1970; Mutis Jurado, 1983) and two of these, Aranzazu and an unnamed occurrence, in Quebrada Mico, near Aguadas, have recently been sampled (Brooks, 2014). The Quindio mercury mine, near Ibague, Dept. Tolima, Colombia may have been worked during ancient time and presumably also by the Europeans (Scheib, 1934; Singewald, 1950). Cinnabar occurrences in Chile have been documented by McAllister et al. (1950) and Ruiz Fuller (1965).

4. Previous Work

The cinnabar-mercury occurrence at Azogues, also referred to as Loma Guashon or Huashun, is approximately 4.5 km east of the town of Azogues (Azogues, 1968). And, even though several geologic references are readily available, the Azogues cinnabar occurrence was dismissed, without field study or thorough review of the literature, as a "red herring" in Andean mineral resource studies by Berger et al. (2016).

Previous work includes:

- A sketch map "Plan del Cerro Mineral de *Azogue* nombrado Guazun (Guashon)" that dates to 1799 shows the adits oriented along the north-south strike of the cinnabar vein (Archivo General de Indias, 1799) (**Figure 1**; **Figure 2**). Whether these mine workings date to Colonial time, or earlier with possible Colonial overprinting, has important implications for regional archaeological and mineral resource studies. Herein, these adits are interpreted to have initially been worked before the arrival of the Europeans. The evidence includes: 1) the numerous adits are shallow and follow the easy-access surface exposures of the cinnabar ore, and similarly, Petersen (1970/2010) indicates that pre-contact underground mining, such as interpreted at Azogues, seldom went for any depth and was typically limited by how far the sunlight could reach into the adit. The absence of mine waste near the entrance also indicates shallow workings. Early mines in Europe rarely penetrated more than 10 m (Craddock, 1995) and the Azogues adits, in comparison, may only reach ~15 m (**Table 1**). Also see **Figure 1** and note the presence of *llumbreras* (described in legend at lower left) that have been interpreted as smaller adits cut to the surface to serve as a light source; 2) there are no remnants of interior beams or supports; 3) pre-contact mining was typically limited to softer rocks such as the sandstones and shales (**Figure 3**) found at Azogues (Egüez, 1993a; 1993b); 4) ancient mines typically followed the veins and ore pockets, and not a compass direction, and therefore, were likely to be sinuous (Craddock, 1995); 5) the floors of ancient mines were seldom horizontal; and 6) the Azogues adits do not have the stone entrance

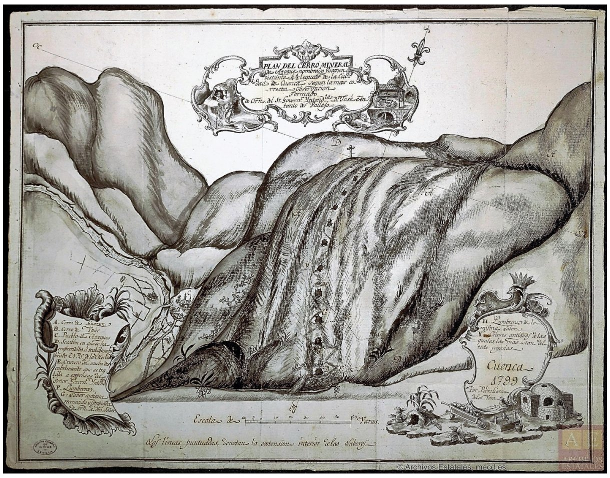


Figure 1. A sketch map of "Cerro Mineral de *Azogue* nombrado Guazun (Guashon)" that dates to 1799 showing adits oriented along the north-south strike of the cinnabar vein (Archivo General de Indias, 1799).

Figure 2. Lowest adit shown in **Figure 1**, note the absence of any entrance architecture.

Figure 3. Sheared, altered sandstone at entrance to adit shown in **Figure 2**.

Table 1. ICP analyses from the cinnabar-mercury occurrence at Azogues (Loma Guashon), Ecuador.

	EC161a	EC161b	EC162a	EC162b	EC163	EC164	EC165	EC166	EC167	TW61
Au (0.003)	0.006	0.006	0.005	0.006	0.005	0.004	na	0.004	na	0.03
Ag (0.2)	2.4	1.5	0.7	0.8	0.7	0.3	0.4	0.4	0.3	–
As (2.0)	75	45	46	43	19	9	8	14	4	1
Ca (1.0)	23,067	1870	24,190	13,707	188,563	2243	11,342	229,571	5379	39,100
Cu (1.0)	89	90	98	96	52	53	71	97	66	–
Fe (10)	55,813	57171	65,646	78,533	55,718	15,762	42,341	52,120	32,532	9800
Hg (0.5)	31.1	33.1	39.2	11.8	4.8	113.3	0.9	1.3	<1	0.03
Mo (1.0)	3	14	7	14	10	<1	<1	<1	<1	0.2
Pb (3.0)	220	143	85	87	60	44	13	46	12	7
S (10)	7262	878	741	417	270	182	289	180	276	240
Sb (0.3)	54	34	22	21	15	10	<1	12	<1	–
U (8)	<8	<8	<8	<8	<8	<8	<8	<8	<8	0.45
Zn (1.0)	371	263	249	228	132	70	105	112	117	–

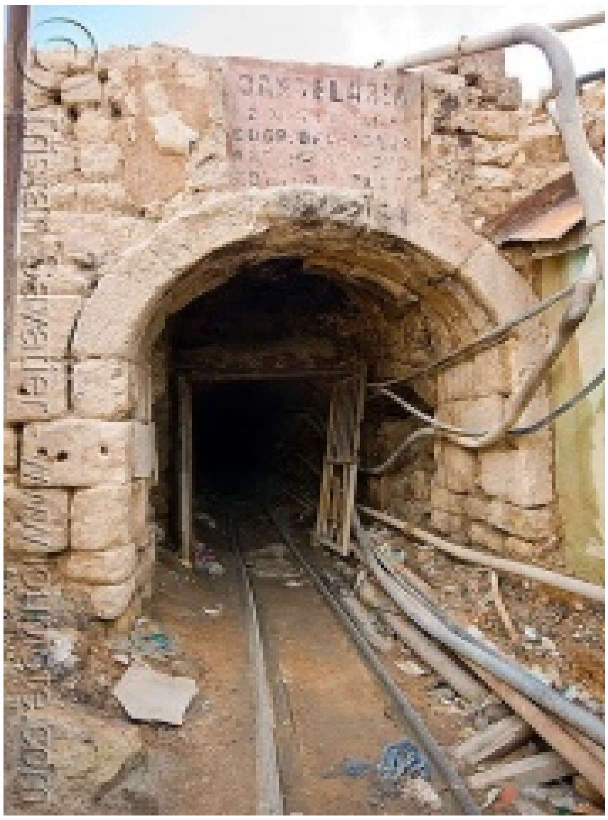
Multi-element ICP analyses (parts per million; detection limit given to right of element, in parentheses; na-not analyzed); American Assay, Sparks, NV, (SP0117075). Sample Descriptions: EC161a [741836/9696463 UTM] area sample inside lowest adit cut into NS striking vein, in rusty hematite altered sandstone, adit is ~15 m, entrance is ~2.2 m by 1.9 m, light comes in at the end of the adit from a stairstep-cut, steep ~40˚, *llumbrera*; quartz-biotite rhyolite in float. EC161b: [741836/9696463] spot sample at entrance, vertical shearing; EC162a: [741645/9696954] area sample ~1 - 2 m within the steep > 40˚, stairstep-cut *llumbrer*a, ~1.5 × 2 m, yellow alteration, jarosite? and hematite, following NS vein; EC162b: [741645/9696954] spot sample at entrance, vertical shearing; EC163: [741651/9696968] grab sample away from adit, minor quartz vein, limonite and hematite staining; EC164: [741615/9696928] south side of road, grab sample, on strike with NS vein and lowest adit, in road cut, quartz veins, white altered clay, rhyolite? EC165: [741343/9696968] soil sample, north side of road, from small terrace with broken tiles and wood; EC166: [741482/9696908] on terrace, sample of sandstone "float" with yellow stain, goethite/hematite, and cross-hatched, cm-sized, Fe-altered pattern; EC167: [741654/9696898] soil sample, dark, 3 - 4 cm deep, south side of road; TW61: Background distribution of elements in sandstone, in ppm; –, not available (Turekian & Wedepohl, 1961).

architecture typical of mines that were exploited mainly during Colonial time, for example, the Santa Barbara mercury mine, Huancavelica, Perú (**Figure 4(a)**, **Figure 4(b)**), or the La Candelaria silver mine, Potosí, Bolivia (**Figure 4(c)**).

- Chacon (1986) discussed Gil Ramirez Davalos' (governor of Quito and founder of Cuenca in 1557) Colonial ownership of the mercury mines at Cerro *Azogue* (Guashon); however, no data on mine production was included. Therefore, Azogues *may* have been a Colonial source of mercury, however, Azogues was a far smaller occurrence and less important than Huancavelica's mercury that was used for silver amalgamation during the late 1500s.
- Wolf (1892/1975) visited the Azogues area and documented "…old mercury mines in the Huaizhun (Guashon)… with large well-worked galleries… but not a drop of native mercury was found… it appeared that the occurrence

(a) (c)



(c)

Figure 4. (a) Colonial stone architecture (interior and right of archway) at entrance to Santa Barbara mercury mine, Huancavelica, Perú; (b) Carved Spanish shield at entrance to Santa Barbara mercury mine, Huancavelica, Perú; (c) Colonial stone architecture, with modern mining equipment, at entrance to La Candelaria silver mine, Potosí, Bolivia (http://www.loupiote.com/photos/5043777416.shtml).

was completely exhausted... however; rock samples sent to Lima gave ½% Hg (or ~5000 ppm Hg)." He also reported that *campesinos* (farm workers) near the mines found *azogue* in the soils and "…mercury droplets were found at Peñas de Guayaquil… at the foot of Cerro Santa Ana… in the Rio Guayas…

and Rio Daule" and he suggested that the mercury was released during small-scale gold mining (pre-contact? or Colonial?) by *lavaderos* (small-scale gold miners) panning in the region.

- The Geologic Map of Ecuador (Egüez, 1993a) shows Cretaceous sandstones and other sedimentary rocks at Azogues and, most importantly, the companion Metallogenic Map of Ecuador (Egüez, 1993b) shows a mine symbol with the number 117 that corresponds to "Hg" in the accompanying list of Names and Details of Metallic Mines included on the map explanation. Delbridge & Robertson (1992) also indicate that Azogues is the only mercury occurrence in Ecuador; however, two other occurrences, Cerro de Camachurco and San Jacinto, are referenced by Chacon (1986) and Vetter Parodi (2016). Other mercury occurrences spatially associated with Ecuador's epithermal porphyry gold-copper systems (Gemuts, 1992; Howell International, 2014) may have been overprinted or removed by recent mining.
- Truhan et al. (2005) researched "…the site of the Colonial, and possibly pre-Columbian, Azogues mines that were found near the municipality of Luis Cordero at Loma Huashún and ancient mining evidence included reddened scoria."

5. ICP Data from Azogues

As noted by Wolf (1892/1975) the Azogues mines had been exhausted of their native mercury; however, rock samples sent to Lima contained 5000 ppm Hg. Therefore, mercury occurrences such as Azogues should be evaluated using modern geochemical methods to document the ore that was produced.

Using the basic tools of mineral resource assessment that include a site visit and geochemical sampling (Inductively Coupled Plasma), the altered sandstones in the adits at Azogues gave 11.8 - 113.3 ppm Hg (**Table 1**), which is consistent with mining that would have long since removed the high-grade cinnabar ore and native mercury leaving only trace amounts of disseminated cinnabar (Duschak & Schuette, 1925) in the wallrock along the north-south structure (**Figure 3**). Craddock (1995) also indicates that Bronze Age miners in Europe were similarly thorough "…and removed every last morsel of ore before a working was abandoned."

Occurrence of the mercury in association with the sedimentary rocks at Azogues is somewhat similar to the geologic setting of Almadén, Spain where the ore is also stratabound, disseminated cinnabar with some native mercury. The geochemical signature at Azogues is much like the geochemical signature at Almadén and Huancavelica (Rytuba, 1986; Noble & Vidal, 1990) and includes As (9 - 75 ppm), Sb (10 - 54 ppm), and other elements of interest such as Ag (0.3 - 2.4 ppm) and Pb (12 - 220 ppm). Background elemental concentration for sandstones is also provided on **Table 1**. The amount of sulfur (182 - 7262 ppm) may be related to the sulfur held in cinnabar; however, other sulfur-bearing minerals may be present. Altered rhyolite was found at one location (**Table 1**, sample

EC164 with 113 ppm Hg); however, there is no field evidence to suggest that Azogues is a hot-spring mercury occurrence.

6. History of Retorting and Ancient Industrial Uses

Retorting cinnabar to obtain mercury dates to 8,000 years ago in ancient Turkey where the 3 m marble base of an ancient retort was found. The metal would have been used for gilding and alluvial gold amalgamation (Barnes & Bailey, 1972; Brooks et al. 2017). Fine-grained gold from the Takht-e Soleyman mining district, northwest Iran, was first panned and then mixed with mercury for retorting (Momenzadeh et al., 2016). And, also in Mesopotamia, ancient cinnabar processing on a smaller scale is indicated by the Tepe Gawra pot (3500 BC) that has been interpreted as an ancient mercury retort (Levey, 1955).

The earliest written description of mercury used for alluvial gold mining was by al-Biruni, an 11th century Persian scientist. Small pits in the Sind (Indus) Riverbed were filled with mercury; the mercury amalgamated the alluvial gold flakes and then the mercury-gold amalgam was recovered, squeezed in a cloth to remove most of the mercury, and then the amalgam "nugget" was burned to volatilize any remaining mercury (al-Hassan & Hill, 1986). This anthropogenic gold "nugget" could then be fashioned into jewelry or hammered to produce gold foil.

In Europe, Agricola (1556/1912) indicates that the use of mercury for gold amalgamation dates at least to Roman times, whereas the use of mercury for silver amalgamation dates only to the 16th century. This chronology is consistent with the onset of the use of mercury from Huancavelica, Perú and mercury imported from Almadén, Spain for silver amalgamation, specifically the Patio Process, during Colonial time in the New World (Barba, 1640/1923; Crozier, 1993; Robins, 2011).

In Perú, ancient retorting is indicated by ceramic retorts (*hornos antiguos*) that date to AD1400 that were found near the Huancavelica mines (Rivero & Tschudi, 1851 in Petersen, 1970/2010). Therefore, retorting cinnabar and the end-uses of the mercury in the ancient Andes, whether from Huancavelica, Azogues, or any of a number of other mercury occurrences, are thought-provoking. And, given the availability of cinnabar and the amount of gold masks, pectorals, and other gold artifacts that were produced before the arrival of the Europeans, then retorting of cinnabar and amalgamation are consistent with any discussion of ancient gold mining and artifact production in the Andes. The mercury produced from the ancient Huancavelica retorts would have had but one use—much as mercury is widely used today—for small-scale alluvial gold mining (Cabrera La Rosa, 1954; Larco Hoyle, 2001; Sandoval, 2001; Cánepa, 2005; Brooks et al. 2013).

7. Human Health

In Perú, Garcilaso de la Vega (1539-1616) commented that mercury fumes were unhealthy and inhalation resulted in "shakes" and loss of other senses (Brown, 2001; Larco Hoyle, 2001)—this observation regarding mercury and human health is very important. The implications are straightforward and provide evidence of 3 possible ways in which the ancient Andean miners and metallurgists would have been exposed to mercury fumes:

1) during ancient cinnabar mining and fire setting—Similar to Garcilaso de la Vega's observations in Perú, modern cinnabar mining at Almadén exposed workers to mercury dust and fumes that affected the nervous system (Putman, 1972). However, because of the smoke, dust, fire, heat, toxic fumes, and sulfurous smell resulting from the ancient method of fire setting, the mine would have been evacuated (Craddock, 1995). The cinnabar recovered from this process would have had two industrial uses: 1) sorted and ground for use as a pigment, or 2) retorted to provide mercury.

2) during ancient cinnabar retorting—Agricola (1556/1912) showed racks of ceramic retorts being fired and also warned of the adverse health effects of breathing the sweet-smelling mercury fumes that were released during retorting. When cinnabar is retorted, the metallic bonds between mercury and sulfur are irreversibly broken and do not recombine to precipitate as cinnabar. Therefore, in the retort in the presence of oxygen, the products would have been sulfur dioxide (gas) and mercury vapor that would have been cooled, condensed, and collected as liquid mercury (Roskill, 1990), as in the following reaction:

$$HgS + O_2 = SO_2 + Hg$$ (Duschak & Schuette, 1925).

However, owing to the imperfections in the ancient ceramics or in the seals between the ceramic vessels, then this process would have released Hg fumes, along with SO_2, from the retorts or up the chimney (Brooks et al. 2017). Any of the pink- to light-red calcined material resulting from retorting may have been plowed under, covered by jungle growth, debris flows, or removed by rainfall. Retorting would have provided mercury to be used for ancient small-scale alluvial gold mining.

3) or from burning (*refogado*) the gold-mercury amalgam—in order to part the alluvial gold from the mercury-gold amalgam "nugget", the "nugget" was burned (*refogado*) with a blowpipe (*soplete*), or in modern times, with a gas torch. This burn would have released dark mercury fumes that modern miners and gold dealers in Madre de Dios or southern coastal Perú, for example, rigidly avoid during amalgam burning (Cánepa, 2005). A Benzoni sketch from the 1500s (Petersen, 1970/2010) shows that their "*sopletes*" are above the flames. This would have concentrated and provided higher oxygen content, which in turn provided the higher temperatures necessary to volatilize the remaining mercury and melt the gold (1063˚C), much as an analytical blowpipe was used for mineral analysis in the 1700 - 1800s. This analytical technique, which dates to 1500 BC and was used by Egyptian goldsmiths, was widely used for analytical chemistry of minerals through the mid-1800s (Dana & Ford, 1922; Jensen, 1986). The Benzoni sketch shows how the workers would have been exposed to the

fumes during the *refogado* process. And, most of the approximately 2 kilograms of mercury required to produce 1 kilogram of gold (Roskill, 1990; Cánepa, 2005) would have been released, as toxic fumes, during the *refogado* process (Brooks et al., 2007; 2013).

Fuel for retorting would have been supplied from the abundant eucalyptus trees in the region or possibly from lignite in the Cuenca-Azogues-Biblian region (O'Rourke, 1978; Weaver & Wood, 1994; Hackley & Brooks, 2006).

8. Conclusion

Cinnabar and mercury were known and used for only two purposes in the pre-Columbian Andes and mercury was essential to Colonial silver processing. Therefore, site visits, geochemical sampling, and assessment of the cinnabar occurrences in the Andean region, such as Azogues, are important to understand the availability of cinnabar and mercury for ancient industrial applications that included: 1) pigment production, and 2) retorting cinnabar to obtain mercury for small-scale gold production. Geochemical sampling, combined with information from published documents, unequivocally indicated the availability of cinnabar and mercury at Azogues (Loma Guashon). The number of shallow workings and geometry of the Azogues adits combined with the lack of stone entrance architecture and other physical evidence at Azogues are consistent with initial pre-contact cinnabar mining for ancient industrial uses such as vermilion production and mercury retorting. However, the absence of Colonial entrance architecture indicates that it was unlikely that the Azogues occurrence was a significant source of Colonial cinnabar.

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