

Lead provenance for medieval decorated tile glazes from Brittany and Anjou (13th-14th c.)

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- 1 TITLE
- 2 Lead provenance for medieval decorated tile glazes from Brittany and Anjou (13th-14th c.)
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103 DECLARATION OF INTEREST

104 Declarations of interest: none.

105

106 ABSTRACT

107 Medieval pavements composed of lead-glazed tiles decorated with a variety of techniques 108 continue to inspire questions about the organisation of glaze manufacture, and the supply and 109 origin of lead materials. The tiles analysed in this study are from Suscinio I (a 13th-century 110 pavement) and Suscinio II (a 14th-century pavement), at the Château of Suscinio in Brittany, and 111 also from the 14th-century pavement at the fortified manor house in Brain-sur-Allonnes, Anjou. 112 Lead isotope analysis (LIA) was used to examine samples from 44 lead-glazed tiles, 29 of which 113 are transparent, while 15 are tin-opacified (an exogenous technique in these regions during this 114 period). Five out of the six LIA groups thus identified favour site-specific supply networks, while 115 results for the remaining group, LIA5, indicate a multi-site supply network. After combining LIA 116 results with archaeological and historical data, the most likely provenance for the lead materials 117 in LIA5 is Derbyshire, in the British Isles. Both the importation of ready-to-use glazing mixtures 118 and the use of lead from neighbouring mines can now therefore be discounted as plausible 119 hypotheses for the production of the tin-opacified lead-glaze tiles analysed in this study.

120

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122	- Two lead supply strategies: site-specific and multi-site
123	- Imported lead supply strategy for both transparent and opaque glazes
124	- Derbyshire is the most plausible lead source for the multi-site supply network
125	- Tin-opacified glazing mixtures were locally produced
126	
127	
128	KEYWORDS
129	Middle Ages; tin-glazed earthenware; high lead glaze; lead isotope analysis (LIA); decorated tile
130	technology; Suscinio (Brittany, France); Brain-sur-Allonnes (Anjou, France)
131	
132	1. INTRODUCTION
133	Decorated tile pavements in religious and secular Gothic buildings throughout north-western
134	Europe reflected the power of ecclesiastical and lay princes (Gauthier, 1984; Crépin-Leblond and
135	Rosen, 2000; Rosen and Crépin-Leblond, 2000). These prestigious floor coverings were almost
136	invariably composed of transparent lead-glazed tiles, decorated using various techniques, all of
137	which seem to have been developed locally. In contrast, tin-opacified lead-glaze tiles, also
138	described as tin-glazed tiles (Norton, 1984b), and termed faïence or carreaux stannifères in
139	French (Norton, 2000), are extremely rare (Bon, 2000; Chapelot, 2000; Démians d'Archimbaud
140	
140	et al., 2000; Jugie, 2000). This technique, originating in the Ummayad and Abassid Caliphates of
140	et al., 2000; Jugie, 2000). This technique, originating in the Ummayad and Abassid Caliphates of Egypt and Western Asia in the 8th to 9th century, spread gradually to the western Mediterranean
140 141 142	et al., 2000; Jugie, 2000). This technique, originating in the Ummayad and Abassid Caliphates of Egypt and Western Asia in the 8th to 9th century, spread gradually to the western Mediterranean in the following centuries (Norton, 1984a, 1984b, 1984c; Matin et al., 2018; Pradell and Molera,

144 A pavement with both types of glaze suggests the simultaneous presence of indigenous and 145 exogenous technical knowledge and cultural traditions. One such pavement in present-day 146 France, Suscinio II (Figure 1), was discovered still in place in the extramural chapel at the 147 Château of Suscinio, a residence of the dukes of Brittany from the 13th to the 15th century 148 (André, 1986a, 1986b, 2003). By combining historical and archaeological data with ceramic 149 provenance and technological studies (Métreau et al., 2017), it has been established that: (1) the 150 Suscinio II tiles were probably not imported as finished products, but rather produced nearby 151 with locally available clayey raw materials; (2) tin-opacified lead glazes do not necessarily 152 indicate local experimentation, but could suggest technical knowledge from further afield.

153



155Figure 1. (a) The medieval pavement of Suscinio II, excavated in 1975 (© G. Chapuy, in Métreau et al., 2015). (b)156The four types of decoration on the lead-glazed tiles (© L. Métreau, adapted from Métreau et al., 2017).

157

In medieval times, lead was used for many purposes (Benoit, 1985; Téreygeol, 2009). Galena
(PbS)—also known by potters as *alquifoux* (Picon et al., 1995)—and litharge (PbO) were two of
the lead compounds frequently used to lower the silica melting point in glazing mixtures (Abel,
1975; Tite et al., 1998). The lead present in archaeomaterials can be characterised by lead isotope

analysis (LIA), a technique that has been used since the 1960s (Grögler et al., 1966; Brill and

163 Wampler, 1967; Gale and Stos-Gale, 2000). Studies using LIA on glazes have generally focused

164 on technological choices and specific distribution networks, as well as on lead sources (Mason et

165 al., 1992; Wolf et al., 2003; Stos-Gale, 2004; Iñañez et al., 2010; Walton and Tite, 2010; Shen et

166 al., 2019; Fornacelli et al., 2020; Medeghini et al., 2020; Chang et al., 2021).

167 In this study, 13 transparent and 13 tin-opacified lead-glaze tiles from Suscinio II, a 14th-century

168 pavement, are investigated through LIA. The results should provide key information about lead

sources and supply strategies, in particular the question of local, regional, or international supply

170 networks, and the hypothesis of imported ready-to-use glazing mixtures to produce the tin-

171 opacified glazes, possibly from regions around the western Mediterranean, in particular the

172 Iberian Peninsula. Although Suscinio II is the focal point of this study, further temporal context is

173 provided by Suscinio I, a 13th-century pavement. Further spatial context is provided by the 14th-

174 century pavement of Brain-sur-Allonnes (Anjou). Exploring the origin, initial production, and use

175 of tin-glazing in Brittany and Anjou will contribute to a better understanding of the long-standing

176 tradition of the French tin-glazed earthenware technique and its place within the history of

177 decorated tiles.

178

179 2. MATERIALS AND METHODS

180 **2.1 Decorated tile pavements**

The tiles are from three different pavements at two archaeological sites (Figure 2): Suscinio I and Suscinio II are pavements from the Château of Suscinio in Brittany; the third pavement is from the fortified manor house at Brain-sur-Allonnes in Anjou. To complement the existing historical and archaeological data for these three pavements, the results of three previous archaeometric studies are compiled in Appendix A.



187

Figure 2. Map of the study area. Stars indicate pavement sites (L. Métreau, adapted from André and Le Pennec, 2003).

- 190
- 191 2.1.1 Suscinio I

192 The remaining traces of the Suscinio I pavement include approximately 1,500 tiles and tile

193 fragments, with many different geometric forms (André et al., 2016). Stylistic evidence indicates

that the pavement may have been laid between 1260 and 1270. Two decorative techniques are

195 identified: plain transparent lead-glazed tiles (ca. 67%), and two-colour transparent lead-glazed

196 tiles (ca. 33%).

197 A lead compound (39–62 wt% PbO) was one of the main components of the glazing mixture,

198 together with silica and clay (Métreau et al., 2012b). Detailed examination of the clay body has

199 identified characteristics similar to those of medieval Breton ceramics, while Brittany and Anjou

- 200 are the most probable sources for the different types of non-calcareous clayey raw materials
- 201 (Appendix A).

The Suscinio I pavement may well have been similar in style to pavements found in and around the Anjou region (André et al., 2016). It was probably discarded between 1320 and 1330, and replaced by Suscinio II (André, 2003).

205

206 2.1.2 Suscinio II

207 The tiles from Suscinio I would have been more difficult to produce and fit together than the

simple, standardised, proportionate geometric shapes used for the Suscinio II pavement,

209 composed of approximately 30,000 tiles. Suscinio II may have been purpose-built or redeployed

210 from another site. Its terminus post quem (1330) was identified by coins found in the clay bed

211 under the pavement (André, 2003; André et al., 2016).

Most of the lead-glazed tiles are transparent (plain, ca. 62%; two-colour, ca. 36%), but some are tin-opacified (plain, ca. 0.5%; painted, ca. 1.5%). The main components of the glazing mixtures

214 were silica and a lead compound (transparent = 50–68 wt% PbO; tin-opacified = 35–

215 45 wt% PbO). Clay was added to the transparent glazing mixture, whereas tin oxide was used to 216 produce the opaque glaze (Métreau et al., 2017). Some of the transparent lead-glazed tiles show 217 signs of tin-oxide contamination, suggesting that both glazing processes took place in the same 218 workshop. Technical flaws on some of the tin-opacified lead-glaze tiles may reflect a poorly 219 controlled firing process. As with Suscinio I, detailed examination of the clay body has identified 220 characteristics similar to those of medieval Breton ceramics. The area around the Château of 221 Suscinio is the most likely source for the non-calcareous clayey raw materials (Appendix A). 222 The tiles from Suscinio II are stylistically similar to decorated tiles found in northern France

223 (e.g. at the Abbey of St Bertin, St Omer), at a distance of over 600 km (André, 2003).

224

225 2.1.3 Brain-sur-Allonnes

The lead-glazed tiles (approximately 14,000 tiles and tile fragments) found at the fortified manor house at Brain-sur-Allonnes have simple, standardised, proportionate geometric shapes (Norton and Clair, 1980; Clair and Lecompte, 1986). They can be divided into three types: a majority of plain tiles (ca. 97%), and some two-colour tiles (ca. 2.5%), both with transparent lead glaze, and a small number of painted tiles, with tin-opacified lead glaze (ca. 0.5%).

231 The main components of the glazing mixtures were silica and a lead compound (transparent =

51-65 wt% PbO; tin-opacified = 44-47 wt% PbO). As with Suscinio II, clay was added to the

transparent glazing mixture, whereas tin oxide was used to produce an opaque glaze (Métreau et

al., 2012a). The poor quality of the tin-opacified tiles probably indicates problems with the firing

235 process. The area around Brain-sur-Allonnes is the most likely source for the non-calcareous

clayey raw materials (Appendix A).

Between 1360 and 1370, the manor house was destroyed by fire, thus providing a *terminus ante quem* for the production of the decorated tiles. Archaeomagnetic dating of their clay body
indicates that they were produced at the end of the 13th century, but the fire may have affected
the signal (Goulpeau, 1988). Technical and stylistic studies, however, suggest that they were
probably from a single workshop that produced tiles for Anjou and Touraine during the first half
of the 14th century (Norton and Clair, 1980; Clair and Lecompte, 1986).

243

244 **2.2. Sample collection and LIA parameters**

The number of tiles available for sampling was limited by the destructive nature of the process. Only tiles with no apparent weathering were selected for analysis. As 15 tin-opacified tiles were available, plain and two-colour transparent lead-glazed tiles were collected from each site in similar proportions (Table 1). These tiles were then transported to the Centre of Physics Applied to Archaeology, which is part of the Institute of Research into Archaeomaterials (IRAMAT- CRP2A, CNRS, Bordeaux Montaigne University), and archaeometric studies were used to
identify zones where the impact of weathering was negligible (Métreau et al. 2012a, 2012b,
2017).

253

		PAVEME	ENT	
DECOR	Suscinio I	Suscinio II	Brain-sur-Allonnes	Total
Transparent				
PLAIN	3	6	5	14
TWO-COLOUR	3	7	5	15
Tin-opacified				
PLAIN	-	3	-	3
PAINTED	-	10	2	12
Total	6	26	12	44

254

Table 1. Distribution of high lead glaze san	iples
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256

257 Before sampling, the glaze surface was cleaned with acetone and rinsed with distilled water. As it 258 proved impossible to chip small fragments from the surface with a sharp scalpel, a 2 mm-259 diameter conical diamond tip (RADIOSPARES) connected to a multifunctional tool with 260 adjustable rotation speed (DREMEL, 300 model) was used to collect powder samples (2-29 mg). 261 The tip was cleaned for five minutes in an ultrasonic bath between samples to avoid 262 contamination. As the glazes are rich in lead (35-68 wt% PbO), a 1 mg sample proved sufficient (Wolf et al., 2003). These powder samples were digested in 100 µl concentrated hydrofluoric acid 263 264 at the Archéologie, Terre, Histoire et Sociétés Laboratory (ARTEHIS, CNRS, MC, University of 265 Burgundy). The residue after evaporation at 105 °C was dissolved in 50 µl concentrated nitric 266 acid to convert fluoride into nitrate, and evaporated again at 105 °C. Lead isotope ratios were 267 then measured using a Nu InstrumentTM MC-ICP-MS and an Apex (TM) desolvating nebulizer at the Laboratory of Isotope Geology (University of Bern). For mass spectrometric analysis, the 268 residue was dissolved in 0.5 M nitric acid, diluted to obtain a signal of ca. 20 pA for ²⁰⁸Pb, and 269

spiked with Tl to correct for instrumental mass fractionation. Mercury interference on mass 204

271 was corrected by measuring ²⁰²Hg. Glaze sample measurements were interspersed with

- 272 measurements of international standard reference material, NIST SRM 981. The measured Pb
- 273 isotopic composition of the reference material (Table 2) overlaps with values reported by

274 Rehkämper and Mezger (2000). Measured values are therefore presented without bias correction.

275

Reference	²⁰⁸ Pb/ ²⁰⁴ Pb	²⁰⁷ Pb/ ²⁰⁴ Pb	²⁰⁶ Pb/ ²⁰⁴ Pb	²⁰⁸ Pb/ ²⁰⁶ Pb	²⁰⁷ Pb/ ²⁰⁶ Pb
Galer and Abouchami, 1998	36.7219 ± 44	15.4963 ± 16	16.9405 ± 15	2.16771 ± 10	0.914750 ± 35
Hirata, 1996	36.6800 ± 210	[15.4856]	16.9311 ± 90	2.16636 ± 82	0.914623 ± 37
Rehkämper and Halliday, 1998	36.6969 ± 128	15.4912 ± 51	16.9364 ± 55	2.16677 ± 14	0.914685 ± 49
Rehkämper and Mezger, 2000	36.7000 ± 23	15.4900 ± 17	16.9366 ± 29	2.16691 ± 29	0.91459 ± 13
Thirlwall, 2000	36.7228 ± 80	15.4956 ± 26	16.9409 ± 22	2.16770 ± 21	0.91469 ± 7
Todt et al., 1996	36.7006 ± 34	15.4891 ± 9	16.9356 ± 7	2.16701 ± 13	0.914585 ± 4
White et al., 2000	36.6825 ± 78	15.4899 ± 39	16.9467 ± 76	2.1646 ± 8	[0.9140]
This study (n=11)	36.7084 ± 28	15.4929 ± 9	16.9395 ± 11	2.16704 ± 8	0.91461 ± 4

276

Table 2. Values of NIST SRM 981 from the literature, compared with values obtained in this study. Analytical error
 (2 standard errors of the mean) refers to the least significant digits. Results in square brackets were calculated from
 the data given in the original publication.

280

281 **2.3 Lead isotope analysis (LIA) interpretation**

The lead isotope results were then compared with an ore reference database, containing 138

283 datasets and data points (Appendix B), to eliminate the least plausible candidates and to highlight

284 potential sources. The ore reference database was compiled for this study from the available

285 geological and archaeological literature, and from the Oxford archaeological lead isotope

database (OXALID), which is freely available online (Stos-Gale and Gale, 2009). It includes lead

- isotope ratios obtained on a variety of minerals, with lead ore as the priority.
- 288 Comparing LIA results for ores and archaeological samples will generally eliminate incompatible
- sources, but unequivocal identification of provenance is rarely possible (Cattin et al., 2010). The
- 290 complexity of geological history may result in vast isotopic fields, with great diversity within the

291 same ore body. By contrast, ore bodies many hundreds of kilometres apart may have similar 292 isotopic compositions, making further discrimination impossible. In addition, ore reference data 293 may be limited in number, even for major mining regions. Old mining works are not always 294 dated; mines may not all have been identified; medieval mines may even have been destroyed 295 (Téreygeol, 2009). Lead ores from different sources may have been mixed together, and lead 296 metal was frequently reused over time (Boni et al., 2000). Mixing of sources can only occur 297 during glaze preparation, because the lead in finished glazes is non-recyclable (Mason et al., 298 1992).

Despite these drawbacks, LIA is considered an effective method when used in conjunction with
historical records and archaeological evidence (Wolf et al. 2003; Stos-Gale and Gale, 2009;
Hauptmann, 2020). This combined approach provides a reliable way to discriminate between
potential sources, and thus validate provenance hypotheses.

303

304 3. RESULTS AND DISCUSSION

After a brief presentation of LIA results, the remaining sections will present archaeological and
 historical arguments regarding glaze manufacture and lead supply strategies, the potential sources
 proposed in this study, and the provenance of the lead used in the tin-opacified glazes.

308

309 **3.1 Lead isotope data**

Table 3 presents the range of values for lead isotope ratios, organised by type of glaze and by decorative technique. Based on these results, six groups can be proposed (LIA1 to LIA6), leaving four outliers. Although LIA2 and LIA3 lie close together, they are treated as separate groups because of the slight difference in isotopic values between plain and two-colour transparent lead-

- 314 glazed tiles. No such logical subdivision is apparent for the data points in LIA5, despite the broad
- 315 range of isotope ratios in this group.

3	1	7

Sample ID and Decor	Body type	PbO (wt%)	LIA group	²⁰⁸ Pb/ ²⁰⁴ Pb	²⁰⁷ Pb/ ²⁰⁴ Pb	²⁰⁶ Pb/ ²⁰⁴ Pb	²⁰⁸ Pb/ ²⁰⁶ Pb	²⁰⁷ Pb/ ²⁰⁶ Pb
SUSCINIO I								
Transparent								
PLAIN								
BDX13257	NC1	43.7 ± 1.1	LIA5	38.428 ± 0.008	15.631 ± 0.003	18.468 ± 0.003	2.08089 ± 0.00026	0.84640 ± 0.00016
BDX13260	NC2	38.7 ± 1.5	LIA5	38.419 ± 0.009	15.630 ± 0.003	18.458 ± 0.004	2.08134 ± 0.00027	0.84677 ± 0.00017
BDX13261	NC3	47.7 ± 1.6	outlier	38.417 ± 0.016	15.638 ± 0.005	18.423 ± 0.005	2.08528 ± 0.00041	0.84882 ± 0.00008
TWO-COLOUR								
BDX13255	NC2	52.5 ± 0.9	LIA5	38.464 ± 0.011	15.643 ± 0.004	18.475 ± 0.004	2.08189 ± 0.00029	0.84669 ± 0.00015
BDX13256	NC2	58.4 ± 1.2	LIA1	38.351 ± 0.010	15.643 ± 0.003	18.309 ± 0.003	2.09462 ± 0.00029	0.85434 ± 0.00015
BDX13258	NC2	61.7 ± 0.7	LIA1	38.355 ± 0.010	15.649 ± 0.003	18.296 ± 0.003	2.09632 ± 0.00030	0.85532 ± 0.00015
SUSCINIO II	_							
Transparent								
PLAIN								
BDX12420	NC3	54.0 ± 2.1	LIA4	38.377 ± 0.010	15.624 ± 0.003	18.391 ± 0.003	2.08685 ± 0.00029	0.84955 ± 0.00015
BDX12421	NC3	55.0 ± 1.4	LIA2	38.356 ± 0.011	15.626 ± 0.003	18.356 ± 0.004	2.08964 ± 0.00031	0.85130 ± 0.00015
BDX12422	NC3	64.2 ± 0.2	LIA4	38.390 ± 0.010	15.628 ± 0.003	18.396 ± 0.003	2.08687 ± 0.00029	0.84956 ± 0.00015
BDX12423	NC3	54.5 ± 1.1	LIA2	38.378 ± 0.008	15.632 ± 0.004	18.363 ± 0.004	2.08994 ± 0.00028	0.85124 ± 0.00017
BDX12424	NC3	56.5 ± 0.7	LIA2	38.372 ± 0.011	15.634 ± 0.004	18.367 ± 0.004	2.08932 ± 0.00030	0.85120 ± 0.00015
BDX12425	NC3	50.1 ± 0.9	LIA4	38.366 ± 0.010	15.620 ± 0.003	18.390 ± 0.003	2.08631 ± 0.00030	0.84942 ± 0.00015
TWO-COLOUR								
BDX12412	NC3	58.8 ± 2.7	LIA4	38.409 ± 0.010	15.631 ± 0.003	18.405 ± 0.003	2.08688 ± 0.00028	0.84928 ± 0.00015
BDX12413	NC3	62.9 ± 0.8	LIA3	38.377 ± 0.010	15.630 ± 0.003	18.369 ± 0.003	2.08912 ± 0.00029	0.85088 ± 0.00015
BDX12415	NC3	54.8 ± 2.2	LIA5	38.449 ± 0.010	15.634 ± 0.003	18.469 ± 0.004	2.08180 ± 0.00029	0.84650 ± 0.00015
BDX12416	NC3	68.0 ± 0.4	LIA4	38.387 ± 0.010	15.626 ± 0.003	18.394 ± 0.003	2.08691 ± 0.00029	0.84951 ± 0.00015
BDX12417	NC3	66.9 ± 0.4	LIA3	38.377 ± 0.011	15.631 ± 0.003	18.371 ± 0.003	2.08899 ± 0.00030	0.85085 ± 0.00015
BDX12418	NC3	65.4 ± 0.8	LIA4	38.387 ± 0.010	15.627 ± 0.003	18.396 ± 0.003	2.08663 ± 0.00028	0.84946 ± 0.00015
BDX12419	NC3	58.1 ± 0.4	outlier	38.474 ± 0.010	15.629 ± 0.003	18.480 ± 0.003	2.08188 ± 0.00029	0.84566 ± 0.00015
Tin-opacified								
PLAIN								
BDX12402	NC3	43.7 ± 1.1	LIA5	38.451 ± 0.009	15.631 ± 0.003	18.465 ± 0.004	2.08248 ± 0.00025	0.84654 ± 0.00016
BDX12408	NC3	40.1 ± 3.1	LIA5	38.434 ± 0.016	15.633 ± 0.005	18.474 ± 0.005	2.08047 ± 0.00041	0.84618 ± 0.00008
BDX12410	NC3	35.8 ± 2.6	LIA5	38.452 ± 0.016	15.637 ± 0.005	18.479 ± 0.005	2.08098 ± 0.00040	0.84624 ± 0.00008
PAINTED								
BDX12398	NC3	36.9 ± 1.6	LIA5	38.465 ± 0.009	15.642 ± 0.003	18.480 ± 0.004	2.08132 ± 0.00030	0.84641 ± 0.00017
BDX12399	NC3	42.0 ± 0.7	LIA5	38.436 ± 0.009	15.632 ± 0.003	18.473 ± 0.003	2.08071 ± 0.00028	0.84621 ± 0.00016
BDX12400	NC3	39.1 ± 2.3	LIA5	38.463 ± 0.016	15.641 ± 0.005	18.479 ± 0.005	2.08139 ± 0.00040	0.84643 ± 0.00008
BDX12401	NC3	39.7 ± 2.2	LIA5	38.428 ± 0.008	15.633 ± 0.003	18.460 ± 0.004	2.08163 ± 0.00027	0.84685 ± 0.00017
BDX12403	NC3	38.1 ± 1.4	LIA5	38.457 ± 0.010	15.642 ± 0.004	18.473 ± 0.004	2.08181 ± 0.00024	0.84673 ± 0.00016
BDX12404	NC3	43.8 ± 1.6	LIA5	38.435 ± 0.008	15.632 ± 0.003	18.475 ± 0.003	2.08035 ± 0.00027	0.84612 ± 0.00017
BDX12405	NC3	35.4 ± 2.5	LIA5	38.448 ± 0.007	15.638 ± 0.003	18.472 ± 0.003	2.08145 ± 0.00025	0.84657 ± 0.00016
BDX12406	NC3	36.2 ± 1.2	LIA5	38.441 ± 0.008	15.635 ± 0.003	18.475 ± 0.004	2.08080 ± 0.00024	0.84630 ± 0.00016
BDX12407	NC3	45.1 ± 2.2	LIA5	38.430 ± 0.016	15.632 ± 0.005	18.468 ± 0.005	2.08089 ± 0.00040	0.84643 ± 0.00008

BDX12409	NC3	40.8 ± 1.8	outlier	38.388 ± 0.009	15.626 ± 0.004	18.385 ± 0.004	2.08803 ± 0.00026	0.84994 ± 0.00016
BRAIN-SUR-ALLONNES								
Transparent								
PLAIN								
BDX14616	NC4	61.7 ± 0.8	LIA6	38.474 ± 0.010	15.631 ± 0.003	18.505 ± 0.003	2.07909 ± 0.00029	0.84469 ± 0.00015
BDX14617	NC4	50.7 ± 1.5	LIA6	38.471 ± 0.010	15.628 ± 0.003	18.504 ± 0.003	2.07909 ± 0.00031	0.84459 ± 0.00015
BDX14618	NC4	54.7 ± 1.4	LIA6	38.471 ± 0.012	15.628 ± 0.004	18.503 ± 0.003	2.07917 ± 0.00035	0.84465 ± 0.00016
BDX14619	NC4	55.0 ± 2.0	LIA6	38.495 ± 0.010	15.634 ± 0.003	18.511 ± 0.003	2.07963 ± 0.00029	0.84460 ± 0.00015
BDX14620	NC4	60.8 ± 0.6	LIA6	38.462 ± 0.010	15.627 ± 0.004	18.501 ± 0.004	2.07902 ± 0.00029	0.84465 ± 0.00017
TWO-COLOUR								
BDX14621	NC4	63.3 ± 1.3	LIA6	38.486 ± 0.008	15.634 ± 0.003	18.510 ± 0.003	2.07923 ± 0.00027	0.84463 ± 0.00016
BDX14622	NC4	65.4 ± 0.8	LIA6	38.479 ± 0.009	15.630 ± 0.004	18.507 ± 0.004	2.07896 ± 0.00029	0.84454 ± 0.00017
BDX14623	NC4	56.4 ± 1.5	LIA6	38.477 ± 0.008	15.630 ± 0.003	18.505 ± 0.004	2.07911 ± 0.00028	0.84464 ± 0.00016
BDX14624	NC4	54.1 ± 0.4	LIA6	38.480 ± 0.009	15.631 ± 0.003	18.509 ± 0.004	2.07902 ± 0.00024	0.84454 ± 0.00016
BDX14625	NC4	57.8 ± 0.5	LIA6	38.486 ± 0.007	15.634 ± 0.003	18.511 ± 0.003	2.07910 ± 0.00024	0.84454 ± 0.00016
Tin-opacified								
PAINTED								
BDX14614	NC4	45.2 ± 1.3	outlier	38.312 ± 0.009	15.627 ± 0.003	18.357 ± 0.004	2.08697 ± 0.00029	0.85123 ± 0.00017
BDX14615	NC4	45.9 ± 1.2	LIA5	38.464 ± 0.007	15.642 ± 0.003	18.480 ± 0.003	2.08123 ± 0.00025	0.84644 ± 0.00016

Table 3. Lead content determined by SEM-EDS in previous studies (Métreau et al. 2012a, 2012b, 2017) and lead isotope ratios for the 44 glaze samples. Analytical error for lead isotope ratios is shown as 2 standard errors of the mean.

321	Figure 3 displays the lead isotopic composition of the samples and their elemental composition:
322	²⁰⁷ Pb/ ²⁰⁴ Pb vs. ²⁰⁶ Pb/ ²⁰⁴ Pb (Figure 3a), ²⁰⁸ Pb/ ²⁰⁶ Pb vs. ²⁰⁷ Pb/ ²⁰⁶ Pb (Figure 3b), ²⁰⁸ Pb/ ²⁰⁴ Pb vs.
323	206 Pb/ 204 Pb (Figure 3c), and SiO ₂ vs. PbO wt% content (Figure 3d). When compared with the lead
324	evolution model of Kramers and Tolstikhin (1997), all the data points plot above the line for the
325	young upper crust (Figure 3a), indicating that the ore deposit had incorporated some lead deriving
326	from older crust. In Figure 3c, as LIA1 (BDX13256 and BDX13258) contains lead enriched in
327	²⁰⁸ Pb derived from ²³² Th, this group now lies to the left of the line representing the young upper
328	crust.

329



S1 - Two-colour transparent lead-glazed tiles △ S2 - Two-colour transparent lead-glazed tiles △ B/A - Two-colour transparent lead-glazed tiles △ S2 - Plainted tin-opacified lead-glaze tiles △ S2 - Plainted tin-opacified lead-glaze tiles △ S2 - Plainted tin-opacified lead-glaze tiles

332 333 334	Figure 3. Lead isotope ratios and elemental composition of glaze samples from S1–Suscinio I, S2–Suscinio II, and B/A–Brain-sur-Allonnes: (a) ²⁰⁷ Pb/ ²⁰⁴ Pb vs. ²⁰⁶ Pb/ ²⁰⁴ Pb; (b) ²⁰⁸ Pb/ ²⁰⁶ Pb vs. ²⁰⁷ Pb/ ²⁰⁶ Pb; (c) ²⁰⁸ Pb/ ²⁰⁴ Pb vs. ²⁰⁶ Pb/ ²⁰⁴ Pb; (d) SiO ₂ vs. PbO wt% concentration. For the tin-opacified lead glazes, the tin oxide content was subtracted from the
335	global composition, which was then recast to 100%. The error may be smaller than the symbols.

337 3.2 Organisation of glaze manufacture and lead supply strategies

338 The six groups identified by LIA may reflect separate production phases, during each of which 339 the glazing mixture was prepared with lead from a specific source, for a limited number of tiles 340 (Table 4).

341

		LIA GROUPS AND PAVEMENTS									
	LIA1	IA1 LIA2 LIA3 LIA4 LIA5 LIA6 Outliers									
DECOR	S1	S2	S2	S2	S1 S2	B/A	B/A	S1	S2 B/A		
Transparent											
PLAIN		3		3	2		5	1		14	
TWO-COLOUR	2		2	3	1 1		5		1	15	
Tin-opacified											
PLAIN					3					3	
PAINTED					9	1			1 1	12	
Total	2	3	2	6	17		10		4	44	

342

345

346 Five groups are site-specific, with transparent lead-glazed tiles only: LIA1 (Suscinio I), LIA2,

347 LIA3, and LIA4 (Suscinio II), and LIA6 (Brain-sur-Allonnes), suggesting that several lead

348 supply networks could be implemented for the same technique. The results for LIA5, however,

349 suggest that a single source could have been used for different decorative techniques. Archival

350 documents indicate that several procurement strategies were used for glaze production,

351 depending on the market availability of lead, or the opening of a nearby mine as work progressed

352 (Bon, 1992, pp. 61–93, 120), and on material provided by sponsors (Berthier and Flouzat, 2009),

- 353 or even by royal patronage (Madeline, 2009).
- 354 Almost all the tin-opacified lead-glaze tiles are found in LIA5, apart from two outliers
- 355 (BDX12409 from Suscinio II, and BDX14614 from Brain-sur-Allonnes). This group also
- 356 includes four transparent tiles, one from Suscinio II, and three from the earlier pavement,
- Suscinio I. The tin-oxide contamination of two transparent glaze samples from Suscinio II, 357

³⁴³ Table 4. Decorative techniques and LIA groups for each pavement: S1-Suscinio I, S2-Suscinio II, and B/A-Brain-344 sur-Allonnes.

BDX12415 (LIA5) and BDX12419 (the closest outlier to LIA5), suggests that these two-colour tiles must therefore have been produced in the same workshop as the tin-opacified lead-glaze tiles (Métreau et al., 2017). Medieval pottery workshops producing a variety of wares are known to have existed in the late 13th century, in the faubourg Sainte-Barbe, Marseille, France, for example (Thiriot, 1995).

363

364 3.3 Potential sources of lead materials

Comparison of the results for the LIA groups with the ore reference database (Appendix B) shows that, for each of the six groups, around three-quarters of the potential sources of lead materials are incompatible. The remaining quarter, whether datasets or data points, can be classified as either possible or plausible sources for the lead used in the glazes (Table 5).

	LIA GROUPS AND PAVEMENTS					
	LIA1 S1	LIA2 S2	LIA3 S2	LIA4 S2	LIA5 S1+S2+B/A	LIA6 B/A
INCOMPATIBLE	99	98	98	93	100	107
COMPATIBLE	37	38	38	41	36	29
Possible	29	30	30	24	26	23
Plausible	8	8	8	17	8	6
Good match					2	

³⁷⁰

- 376 sources. To evaluate potentially compatible sources for the lead in the glazes, relevant
- archaeological and historical arguments must be explored. For this purpose, the ore reference

Table 5. Degree of compatibility between the database (Appendix B), LIA groups, and pavements.

Possible sources present some drawbacks, because the dataset contains outliers, consists of a

³⁷⁴ small number of data points, or covers a broad isotopic field. Plausible sources are more likely

³⁷⁵ candidates, with a narrow isotopic field. Good matches potentially indicate the most likely

378 database (Appendix B) was organised into six major geographic zones, further subdivided into 379 34 subzones (Figure 4).



380

381 382 Figure 4. Map of the ore reference database (Appendix B); circle, subzone: Zone 1: 1-Armorican Massif, 2-Seuil du Poitou, <u>3</u>-Massif Central; Zone 2: <u>4</u>-West Asturian Leonese, <u>5</u>-Cantabrian, <u>6</u>-Cantabrian-Basque, <u>7</u>-Pyrenees, <u>8</u>-383 Catalonian Coastal Range, 9-Central Iberian, 10-South Portuguese, 11-Ossa Morena, 12-Betic Cordillera, 13-Southeast Volcanic Province; **Zone 3**: <u>14</u>-Balearic Islands, <u>15</u>-Corsica, <u>16</u>-Sardinia, <u>17</u>-Elba; **Zone 4**: <u>18</u>-Western Alps, <u>19</u>-Central Alps, <u>20</u>-Southern Alps, <u>21</u>-Carnic Alps, <u>22</u>-Ligurian Apennines, <u>23</u>-Apuane Alps, <u>24</u>-Modenese Apennines, <u>25</u>-Southern Tuscany; **Zone 5**: <u>26</u>-Western Rhenish Massif <u>27</u>-Vosges; **Zone 6**: <u>28</u>-Ireland, <u>29</u>-Isle of 384 385

Man, <u>30</u>-Scotland; <u>31</u>-Northern Pennines, <u>32</u>-Southern Pennines, <u>33</u>-The West, <u>34</u>-South West England; <u>pickaxes</u>,
 medieval mining site: <u>BO</u>-Brandes-en-Oisans, <u>G</u>-Goppenstein, <u>LP</u>-Los Pedroches, <u>M</u>-Melle, <u>StL</u>-Saint-Laurent-le Minier; star, archaeological site: <u>S1+S2</u>-Suscinio, <u>B/A</u>-Brain-sur-Allonnes, <u>A</u>-Albarracín, <u>B</u>-Barcelona, <u>Ma</u> Marseille, <u>P</u>-Poitiers, <u>StO</u>-St Omer, <u>S</u>-Seville, <u>U</u>-Utrecht, <u>V</u>-Valencia.

391

392 The pavement sites are all located in Zone 1, which will be examined in greater depth in 393 Section 3.4. Zone 2, discussed in Section 3.5, represents a viable provenance hypothesis for the 394 tin-opacified glazes (LIA5), as this Islamic tradition spread to the southern and eastern parts of 395 the Iberian Peninsula during the medieval period (Caiger-Smith, 1985). No datasets in Zone 3 are 396 plausible sources of lead for the glazes in any of the LIA groups. In Zone 4, although some data 397 points for Goppenstein (in Subzone 19) are compatible sources of lead for the glazes in LIA5, 398 and even LIA1 and LIA6, there is no archaeological or historical evidence to confirm the 399 exploitation of this medieval lead mine before 1474 (Guénette-Beck, 2005; 2019). Two data 400 points in Col du Lautaret (Subzone 18), about 30 km from the medieval mining district of 401 Brandes-en-Oisans (Bailly-Maître and Bruno-Dupraz, 1994), are compatible sources of lead for 402 the glazes in LIA6. In Zone 5, only four datasets in the Western Rhenish Massif (Subzone 26) are 403 plausible sources of lead for the glazes in LIA4 (Appendix B). Zone 6, presented in detail in 404 Section 3.6, provides the most promising results, with plausible sources of lead for the glazes in 405 all six LIA groups, and two good matches for LIA5 (Table 6). The discussion of potential sources 406 for the lead in the glazes will now focus on Zones 1, 2, and 6, illustrated by figures plotting the 407 LIA groups against the relevant lead isotope datasets (see also the maps in Appendix B). 408

	LIA GROUPS AND PAVEMENTS					
	LIA1	LIA2	LIA3	LIA4	LIA5	LIA6
Sources	S1	S2	S2	S2	S1+S2+B/A	B/A
PLAUSIBLE						
Zone 1	•	•	•	•	•	
Zone 2				•	•	•
Zone 4	•				•	•
Zone 5				•		
Zone 6	•	•	•	•	•	•

	GOOD MATCH Zone 6 SUBZONE 32. DERBYSHIRE
409	SUBZONE 34. AVON
410	Table 6. Zone compatibility between lead sources, LIA groups, and pavements. No datasets in Zone 3 are plausible
411	sources.
412	
413	3.4 The local or regional source hypothesis (Zone 1)
414	Archival documents (1384–1386) attest that lead for some of the tin-opacified lead-glaze tiles
415	was mined at a distance of 45 km from the Palace of Poitiers, for the Duke de Berry (Bon, 1992).
416	The local provenance hypothesis for Suscinio I and II would therefore be the Armorican Massif
417	(Subzone 1), where mines for various metals have been exploited since the Bronze Age.
418	Medieval mining activity nevertheless remains difficult to evaluate (Domergue et al., 2006;
419	Le Carlier de Veslud and Jouanet-Aldous, 2015; Le Carlier de Veslud et al., 2017).
420	Figure 5 presents detailed results for Subzone 1, divided into three categories. Although nine
421	plausible matches are identified (Appendix B), the error margin is greater than the match, making
422	these sources appear less likely. Another potential drawback is that there is often only one data
423	point for each ore body in the Armorican Massif, so that comparison between LIA groups and the
424	database is less robust.
425	The only plausible neighbouring source (less than 50 km from Suscinio) is Plumelin, a data point
426	that matches LIA4. The lead isotopic data for LIA2, LIA3, and LIA4 (Suscinio II) are in the same
427	range as the ore bodies south-west of Pontivy, at a distance of about 75 km, while La Villeneuve,
428	about 80 km from Suscinio, is the only dataset compatible with LIA5. Huelgoat, a lead mine
429	about 120 km north-west of Suscinio, which may have been exploited earlier than 1540 (Coativy,
430	2006), is a plausible match for LIA1 (Suscinio I), but with only one data point. Plélauff, a much

431 earlier mine about 100 km away, is a plausible match for LIA2 (Suscinio II), but again with only



433







437 At a distance of 145 km from Brain-sur-Allonnes, Melle (in Subzone 2) presents a possible

- 438 match, but this mine was only exploited from the 6th to the 10th century (Téreygeol et al., 2010).
- 439 Eleven of the transparent lead-glaze samples from Suscino II (LIA2, LIA3, and LIA4) present a

plausible match with Saint-Laurent-le-Minier (in Subzone 3), much further south in the MassifCentral (Appendix B).

442

443 **3.5 The imported glazing mixture hypothesis (Zone 2)**

A fundamental hypothesis at the beginning of this study was the importation from the Iberian
Peninsula of ready-to-use glazing mixtures for the tin-opacified lead glazes. Comparison with the
database shows that LIA5 coincides with a sparsely populated area in the isotopic field of Zone 2
(Appendix B). The Los Pedroches Batholith complex (in Subzone 9) is the only plausible source
(Figure 6), but most of the compatible lead isotope data come from copper ores (Klein et al.,
2009). Furthermore, Grañeda Miñión (2008) indicates that mining activity at Los Pedroches,
which flourished during the Caliphal period, had already collapsed during the early 12th century.

451



Figure 6 (a). Data for Zone 2, showing LIA groups; (b) Subzone 9, data from Los Pedroches, showing LIA groups (Appendix B).

- 456 Norton (1984b) put forward the idea that a tiler from a workshop in the Garonne valley might
- 457 have imported the tin-glazed technique after working at the Abbey of Santas Creus near

458 Barcelona. Although tin-opacified lead glazes were probably used to decorate ceramics in 459 Catalonia from the 14th century (Iñañez, 2007), no tin-opacified lead-glaze tiles are described at 460 the Abbey of Santas Creus (Norton, 1984b, fig. 2, p. 137). The isotopic field of the Catalonian 461 Coastal Range (Subzone 8), extended by one uncharacteristic data point, is not coherent with 462 LIA5, nor any of the other LIA groups. Workshops using the tin-glazed earthenware technique, 463 mainly for the production of ceramics, are known to have existed from the 12th century, further 464 south, near Valencia (Manises and Paterna) and Seville (Iñañez et al., 2008). However, no 465 plausible match for any of the LIA groups exists with the isotopic fields of the Betic Cordillera 466 (Subzone 12) or the South-East Volcanic Province (Subzone 13). The LIA study of four 11th-467 century tin-opacified lead-glaze ceramics from Albarracín (Marzo et al., 2009) produced results 468 similar to those for LIA5, but no specific Iberian provenance could be identified.

469

470 **3.6** The most plausible hypothesis: lead of British origin (Zone 6)

Comparison between LIA groups and the database identifies Zone 6 as the most plausible
provenance for all the lead glazes sampled (Appendix B). The Isle of Man (Subzone 29) is a
plausible source for LIA1. In Subzone 31, there are two plausible sources, Cumbria for LIA1 to
LIA4, and Durham for LIA4. In Subzone 32, Cheshire is a plausible source for LIA4, LIA5, and
LIA6. Subzone 33 is also a plausible source: the Shropshire Hills only for LIA1, Clwyd for all
groups except LIA6, and Gwyned for all six groups. In Subzone 34, the plausible sources are
Cornwall for LIA2 to LIA5, and Somerset for LIA4 (Figure 7).



480 Figure 7. LIA groups (black ellipses) plotted against ore data from Zone 6 (Appendix B).481

482 Results for Zone 6 also indicate two good matches, Avon and Derbyshire, as potential 483 provenance for LIA5 (Appendix B, Figure 7). A British origin for the lead in the tin-opacified glazes challenges two previously explored hypotheses: the opening of mines in neighbouring 484 485 regions (Section 3.4; Figure 5), and the importation of ready-to-use glazing mixtures from the 486 Iberian Peninsula (Section 3.5; Figure 6). At the turn of the 14th century, Derbyshire was the main lead-producing area in England and Wales, exporting lead to Northern Europe, mainly via 487 488 Hanseatic trade routes (Blanchard, 2005), and thus appears to be a more historically plausible 489 solution than Avon. Both Suscinio, on the coast of Brittany, and Brain-sur-Allonnes, in the Loire 490 Valley, had easy access to Atlantic trade routes. Stylistic and technical affinities between 491 Suscinio II and contemporaneous Flemish pavements suggest other routes, along the English 492 Channel, to the Abbey of St Bertin, in St Omer, and to sites around Utrecht, where a workshop 493 producing tin-opacified lead-glaze tiles has been identified (de Groot and Pot, 1985).

494 The quality, accessibility, and economy of English tin might explain why lead from foreign 495 sources was preferred (Muhly, 1985; Tylecote, 1987). Piccolpasso's 16th-century treatise on 496 Italian maiolica (1556-1557) mentions that tin from Flanders was recommended for opacifying 497 lead glazes, but the source of this tin was south-west England (Lhôte, 2007; McSweeney, 2011). 498 The use of an oxidised lead-tin alloy (termed *calcine*) ensures homogeneous distribution of the 499 opacifying tin oxide (SnO₂) throughout the glaze. To act as an opacifier, the tin-oxide content of 500 tin-opacified lead glazes should be at least 5 wt% SnO₂ (Mason and Tite, 1997). A tin-oxide 501 content ranging from 20 to 25 wt% SnO₂ has been observed on high-quality Mediterranean wares 502 (Picon et al., 1995). A ready-to-use lead-tin alloy thus seems very unlikely, since the craftsmen 503 would not be able to control the quality of the *calcine*. For the tin-opacified lead-glaze tiles of 504 Suscinio II, a single supply route from England for both lead and tin (possibly in the form of 505 ingots), thus appears to be the most plausible hypothesis.

506

507 4. CONCLUDING REMARKS AND PERSPECTIVES

508 This exploratory study will contribute to the growing body of knowledge about the history of 509 decorated tile pavements, and the tin-glazed earthenware technique, or *faïence*. The corpus of 510 44 tiles, representative of three medieval pavements and four decorative techniques, is large 511 enough to produce meaningful results. Lead isotope analysis of glaze samples, when combined 512 with archaeological and historical evidence, can provide important information about lead supply 513 networks, notably the provenance of the lead in the tin-opacified glazes from Suscinio II and 514 Brain-sur-Allonnes (14th century). The plausible matches between Zone 6 and all the LIA 515 groups, and the good match with English lead for LIA5, together suggest a rather unexpected 516 supply route. As the same lead was used for the tin-opacified glazes, and for some of the 517 transparent lead-glazed tiles from Suscinio, the Derbyshire provenance for LIA5 tends to

518 discount the hypothesis of an imported ready-to-use glazing mixture for the opaque tin glazes. 519 The positive identification of the British Isles as a source of imported lead could be the starting 520 point for future investigations of other medieval tin-opacified lead-glaze products. At other sites, 521 written sources may have been preserved that could provide more information about the 522 motivations underlying the choice of specific types of lead for such products. Identifying the 523 supply routes for both lead and tin is among the ultimate goals for such studies. 524 525 **ACKNOWLEDGEMENTS** 526 The authors would like to thank the following colleagues, whose assistance and judicious 527 comments made this study possible: Françoise Bechtel, Adelphine Bonneau, Michel Boucher-528 Bredoux (A.R.E.G.H.A.T), Peter Davey, Joseph Gauthier, Bernard Gratuze, Barbara Guénette-529 Beck, Fanny Madeline, Fabrice Monna, Jean Rosen, Florian Téreygeol, and Sophie Wolf. The 530 revised manuscript benefitted from constructive suggestions by two anonymous reviewers, whose 531 help is gratefully acknowledged. 532 533 **FUNDING SOURCES** 534 Financial support for data collection, analysis, and interpretation, together with the writing of the 535 report, and the decision to submit the article for publication was provided by the following 536 bodies: Bordeaux Montaigne University, the French Ministry of Higher Education and Research 537 (MESR), the Centre of Physics Applied to Archaeology at the Institute of Research into

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