

WHITE EARTHENWARE FROM LORRAINE (1755 – c. 1820): PROVENANCE AND TECHNIQUE*

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Fragments of 25 examples of 'white earthenware' from Lorraine were subjected to porosity analysis, X-ray fluorescence analysis, X-ray diffraction analysis and backscattered-electron image analysis—coupled with energy-dispersive spectrometry to determine the porosity, bulk, major, minor and trace element compositions, and the composition and the proportion of their constituent phases. These objects embrace two distinct types of paste, CaO-rich and CaO-poor. Both are porous. They are artificial pastes obtained by mixing an imported refractory white firing clay with a temper (calcined flint or sand, crushed biscuit) and a flux (chalk, frit) for the CaO-rich, and without flux for the CaO-poor bodies. Calculated body recipes are comparable to those published in contemporaneous manuscripts. The CaO-rich bodies are covered either with an opaque stanniferous white glaze or with a high-lead glaze, the CaO-poor only with the latter. Maximum firing temperatures were 950–1050°C for the first group, and higher, but not exceeding 1200°C, for the second group. The productions from Lunéville and Saint-Clément can be recognized by their chemical composition, which enables us to confirm or reject attributions based on purely stylistic arguments.

KEYWORDS: TERRE DE PIPE, TERRE ANGLAISE, WHITE EARTHENWARE, LUNÉVILLE, SAINT-CLÉMENT, CHEMISTRY, MINERALOGY, TECHNIQUE

INTRODUCTION

This paper deals with a particular class of ceramics made up of a synthetic and porous paste that turns white during firing and is covered with a glaze, except for on the figurines. In English, it is called *creamware* (Majewski and O'Brien 1987), in German *Steingut*, in Italian *terraglia* and in French *faïence fine*. For several reasons that are discussed later on, we prefer to call it simply 'white earthenware', a neutral term.

A brief history of the invention of 'white earthenware' in England and France

In England, in around 1725, Thomas and John Astbury began to produce white salt glazed stoneware from white clays, and Thomas Whieldon and Josiah Wedgwood obtained white creamware between 1754 and 1759. In France, the history of the *terre de pipe* at the beginning of the 18th century is closely linked with that of soft paste porcelain (Maire 2008). On 30 July 1743, Claude-Imbert Gérin was granted a 10-year privilege to found, in Paris, a royal manufacture of *faïence imitating that of England, the composition of which is made with a different clay from the one used to make ordinary faïence*, which was established in the rue de Charenton.

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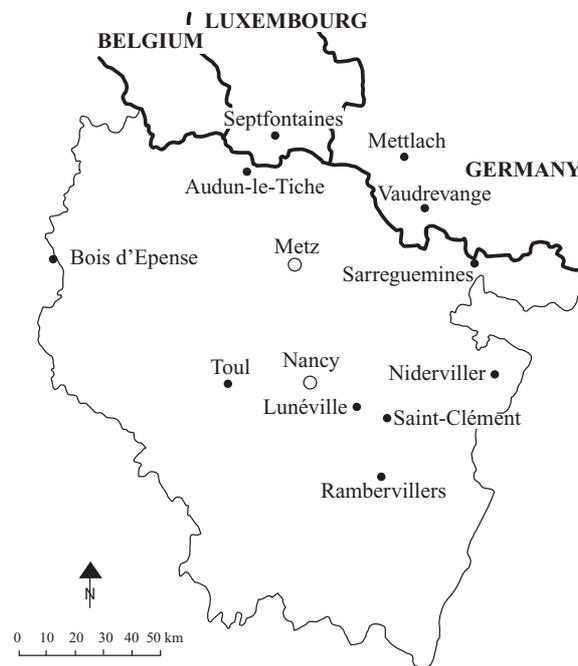


Figure 1 A map of Lorraine, showing the locations of places mentioned in the text. Open circles, major towns; solid dots, manufactures.

In 1745, because of the special properties of the local white clay in Montereau, François Mazois had become associated there with John Hill and Jacques Chapelle, *to work the clay after the English manner, as they are produced in the faubourg Saint Antoine*. The rue de Charenton manufacture was transferred in 1749 to a larger place near the *Pont-aux-Choux* in Paris. In Lorraine (Fig. 1), Jacques II Chambrette managed to produce white earthenware in Lunéville a few years prior to 1748, perhaps around 1731 (Peiffer 2007, 124). All in all, Peiffer counted 23 Lorraine manufactures likely to have produced whiteware in the 18th century (Peiffer 2007, 126; see also Maire 2008).

Nomenclature

French nomenclature in the 18th and 19th centuries In these centuries, the names and classification of 'white earthenware' were complex, often contradictory and covered a wide range of terms such as 'fine pottery', 'white faïence', 'English earthenware', *terre de pipe*, 'English faïence', 'white earthenware' and 'cailloutage' (M. O*** 1807; Société d'Encouragement 1829; Bastenaire-Daudenart 1830). The first edition of Brongniart (1844) classified 'white earthenware' (*faïence fine*) into three types: (1) the so-called *terre de pipe* (*faïence fine marnée*), with a calcareous body; (2) the *creamware* or *cream-coloured earthenware* (*faïence fine cailloutée, cailloutage, terre anglaise, poterie façon anglaise*), with a non-calcareous body; and (3) the 'semi porcelain' (*porcelaine opaque, ironstone, faïence fine dure, faïence fine feldspathique, lithocérame*).

Coloured	Whitish
Coloured earthenware	White earthenware
Stoneware	Porcelain

Figure 2 A modern classification of fine grained, clay based ceramic objects according to body colour and porosity, measured by water adsorption. Earthenwares have water adsorption higher than 2%.

Modern classification Clay-derived ceramic objects with a white body belong either to the family of the ‘white earthenware’, if they have a water adsorption >2%, or to the *porcelains*, if their water adsorption is <2% (Salmang and Scholze 1968, table 45) (see Fig. 2). Today, four types of ‘white earthenware’ are distinguished: (1) ‘calcareous white earthenware’; (2) ‘siliceous white earthenware’; (3) ‘feldspathic white earthenware’; and (4) ‘mixed white earthenware’, which contains chalk and feldspar as fluxing elements. The first type is also called ‘soft white earthenware’, and the others ‘hard white earthenware’. As the classical ‘creamware’ belongs to the second group, a term including the four categories had to be found. This is the reason why we use the general term ‘white earthenware’ (*terres blanches* in French).

Technique of ‘white earthenware’ in France (18th–19th centuries)

Manuscript sources of the 18th century Jean-François Boch gives the recipes used by the workshops of Audun-le-Tiche (Lorraine) and of Septfontaines (Luxembourg), as well as a transcription for that at Saint-Clément (Peiffer 2003, 44; Maire 2008, 23) (see Table 1). Unfortunately, the unit of measurement is not always indicated. The manufacture of Aprey (Central France) used ‘Cologne clay’ in around 1769, as well as Troyes chalk (Delvaux 1908). Joseph de Boussebart specifies in 1786, for the Liège manufacture, that three ingredients are necessary to make a good ‘calcareous white earthenware’ (*terre de pipe*): a ball clay that turns white on firing, flint with the same property and white chalk. The clay comes from Vallendar (Westerwald), a clay bed near Koblenz on the right bank of the River Rhine, and is despatched from Cologne. The technique that Boussebart describes is very similar to that of faïence (Bastenaire-Daudenart 1828). The formula for the recipe that Jacques II Chambrette used in his Lunéville manufacture has not been published as yet (Calame 2009, 54). Lunéville and Saint-Clément used a clay called ‘Cologne clay’, which obviously has nothing to do with the place of extraction, but with the point of sale (Boussebart 1786). The provenance of the raw materials used in some French manufactures in the years 1805–1810 is reported by Dubus and Pannequin (1999). To make the comparison of the recipes in Table 1 easier, the gross values have been recalculated in wt%, supposing that all the original values were in this unit (either in lb or in kg). From these new numbers, five trends seem to emerge: (1) the oldest recipes have no grog or ceramoclast (Ionescu and Ghergari 2002; Hoeck *et al.* 2009); (2) towards the 19th century, the grog replaces part of the flint as inert matter; (3) the earlier frits are alkaline, while the later frits are plumbiferous; (4) the proportions of clay and frit vary considerably (35–52 wt% with respect to 4–25 wt%); and (5) the added chalk remains rather constant (18–23 wt%).

The technical handbooks of the first half of the 19th century The oldest of these technical texts is the anonymous book by M. O.*** (1807), attributed to Oppenheim (Brongniart 1844). He, Boyer (1827) and Bastenaire-Daudenart (1830) give detailed accounts of how to produce *poterie blanche, façon anglaise*; that is, ‘siliceous white earthenware’ in modern terminology. Boudon de Saint-Amans (Société d’Encouragement 1829) presents a thorough explanation of the English

Table 1 Recipes for 'calcareous white earthenware', according to manuscript sources, and calculated porosity-free recipes of selected samples: 1, Audun-le-Tiche; 2, Saint-Clément; 3, Aprey; 4, Septfontaines; 5, Liège; 6, Lunéville; 7, Saint-Clément. Sources: 1, Peiffer (2003); 2-4, Maire (2008); 5, Boussemart (1786); 6, 7, Calame (2009)

	Recipes, raw data							Recipes, recalculated data						
	1	2	3	4	5	6	7	1	2	3	4	5	6	7
Date	c. 1760	1764?	c. 1769	c. 1786	1786	19th century	19th century	1	2	3	4	5	6	7
Unit	?	?	lb	?	lb	kg	kg							
Clay	17	20	17	58	320	275	45	34.0	35.2	35.5	44.6	35.7	43.6	45.0
Flint (calcined)	9	12	10	42	112	100	?	18.0	21.0	20.8	32.3	12.5	15.8	?
Flint	9	12	9	24	208	124	?	18.0	21.0	18.7	18.5	23.2	19.7	18.0
Chalk	15	13	12	6			13	30.0	22.8	25.0	4.6			13.0
Frit 1 (40 flint + 9 sand + 30 soda)														
Frit 2 (unspecified composition)														
Frit 3 (60 calcined flint + 10 white sand + 30 soda)														
Frit 4 (360 calcined flint + 40 sand + 120 soda)					160							17.9		
Frit 5 (100 flint + 9 minium)						62							9.8	
Frit 6 (100 sand from Badonviller + 9 minium)						20							3.2	
Frit 7 (100 flint + 10 salt + 10 minium)							19							19.0
Grog					96	50	5						10.7	5.0
Total	50	57	48	130	896	631	100	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Calculated recipe without porosity (vol %)														
	1	2	3	4	5	6	7							
Clay	TBL 3	TBL 14	TBL 15	TBL 20	TBL 21	TBL 22	TBL 23							
Flint	68.1	61.8	71.7	67.7	72.8	69.0	75.7							
Flint	25.2	28.9	13.2	13.9	14.2	17.7	6.9							
Calcite	6.7	6.6	9.8	9.9	9.6	10.0	10.6							
Frit			5.4	8.6	3.4	3.3	6.9							
Grog		2.6												
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0							

technique for this kind of ware. According to Brongniart (1844), his three types of ‘white earthenware’ are artificial pastes, obtained by mixing: (1) one or more plastic materials such as kaolin or China clay, or highly plastic, kaolinitic and refractory clays (ball clays); (2) one or more non-plastic materials, such as calcined and finely milled flint, quartz and quartz-rich sands or ground fired clay (= grog); and (3) fluxing materials, such as lime, chalk, limestone, dolomite, giobertite (magnesite $MgCO_3$), feldspar, Cornish stone, pegmatite, frit, glass, fusible sand and bone ash. The fluxing materials of the *terre de pipe* are lime or chalk, and feldspar for the third type. He only mentions Lunéville, Mettlach, Sarreguemines, Saint-Clément and Vaudrevange as the places where ‘calcareous white earthenware’ is produced. Surprisingly, one single recipe for such a paste—but Brongniart does not say where it comes from—contains very little calcium: 85.4 parts of clay (75 wt% SiO_2 , 25 wt% Al_2O_3) + 13 parts of flint + 1.6 parts of lime. Although he goes into great detail as far as the process used in Mettlach, Sarreguemines and Vaudrevange is concerned, he does not say anything about that used in Lunéville and Saint-Clément. He writes that Sarreguemines stopped manufacturing ‘calcareous white earthenware’, making only different types of ‘siliceous white earthenware’. The plastic clay that turns white on firing, used here as at Mettlach and Vaudrevange, comes from the Palatinate. Interestingly, none of these textbooks indicates the recipes used in Lorraine for the ‘siliceous white earthenware’. Were they identical to the recipe(s) of the ‘siliceous white earthenware’ manufactures around Paris, where plastic clay from Montereau and calcined flint were mixed in the proportions 88–87 and 12–13 (M. O*** 1807; Bastenaire-Daudenart 1830; Brongniart 1844)?

Issues

A critical reading of the ancient literature shows that if we know nearly all the detailed procedures for the making of the ‘siliceous white earthenware’ in the French manufactures outside Lorraine in the 18th and 19th centuries, we know practically nothing about the way this kind of ‘white earthenware’ and the ‘calcareous white earthenware’ were produced in Lorraine, the French province where the latter is supposed to have been invented by Jacques II Chambrette a few years prior to 1748. Another major problem is how to differentiate between the various Lorraine productions through a purely stylistic approach. The purpose of this study is therefore to yield elements that enable us to answer the following questions:

- (1) Is it possible to differentiate the productions of Lunéville from those of Saint-Clément and other centres from a mineralogical and chemical point of view?
- (2) What is the process used in the preparation of the paste for calcareous as well as siliceous Lorraine ‘white earthenware’?
- (3) What is the process used in the preparation of the glazes?

EXPERIMENTAL

Samples

Twenty-five fragments from various private and museum collections were sampled, with special emphasis on objects believed to have originated in the manufactures of Lunéville and Saint-Clément (Table 2). As to those from the collections of the Castle of Lunéville, which suffered great damage in the blaze of 2 January 2003, we tried to choose fragments with the least possible damage. The studied pieces were selected because most of them could reasonably be attributed to a Lorraine workshop, either by iconographic evidence, the archives of the museum having also

Table 2. A list of the specimens analysed

An. no.	Object	Date	Remarks	Attribution
TBL 1	Teapot	End of 18th century	Embossed decoration, Lorraine <i>terre de pipe</i> , private collection	Lorraine
TBL 2	Bowl with handles	c. 1820	Pink 'lustre Burgos' rosé, stamped Sarreguemines, private collection	Sarreguemines
TBL 3	Figurine	c. 1770	Third-firing polychrome decoration, private collection	Niderviller
TBL 4	Broth bowl with handles	c. 1770	Embossed decoration, Lorraine <i>terre de pipe</i> , private collection	Rambervillers?
TBL 6	Herbal teapot	End of 18th century	Third-firing embossed decoration, <i>terre de pipe</i> , private collection	Lunéville or Rambervillers
TBL 9	Gravy boat dish	End of 18th century	Flat-bottomed, embossed 'grains of rice' pattern, excavation Autun 506, Autun	Lorraine
TBL 10	Fragment	End of 18th century	<i>Terre de pipe</i> , embossed pattern, excavation Autun 538, private collection	Lorraine
TBL 11	Bowl handle	End of 18th century	<i>Terre de pipe</i> , embossed pattern, excavation Autun 546, private collection	Lorraine
TBL 12	Cup	End of 18th century	<i>Terre de pipe</i> , embossed pattern, excavation Autun 547, private collection	Lorraine
TBL 13	Figurine of a woman showing a marmot	End of 18th century	Third firing polychrome decoration, private collection	Bois d'Epense?
TBL 14	Quail	c. 1805	Biscuit, stamped Sarreguemines Utzschneider, Musée de la faïence Sarreguemines	Sarreguemines
TBL 15	Watch case and inkstand	1755–60	Third-firing polychrome decoration, lid decorated with a reclining Chinese figure, arms of de La Gaffelière, similar object in Céramique lorraine (1990, 179), Lunéville castle museum	Lunéville (Chambrette)
TBL 16	Planter?	End of 18th century	Third-firing polychrome and gilded decoration, Céramique lorraine (1990, 132), Lunéville castle museum	St. Clément
TBL 18	Dish	End of 18th cent.	Embossed decoration, Lunéville castle museum	Lorraine
TBL 19	Broth bowl with handles	End of 18th cent.	Embossed decoration, Lunéville castle museum	Lorraine
TBL 20	Pot pourri	c. 1770	Third-firing polychrome and gilded decoration, Lunéville castle museum	Lorraine (Lunéville?)
TBL 21	Watch stand	c. 1770	Third-firing polychrome and gilded decoration, Céramique lorraine (1990, 146), Lunéville castle museum	Saint-Clément
TBL 22	Pot pourri	18th century	Third-firing polychrome, Lunéville castle museum	Saint-Clément
TBL 23	Rectangular bulb planter	c. 1770	Third-firing polychrome, Lunéville castle museum	Saint-Clément
TBL 30	Saucer	End of 18th century	Embossed decoration, Lunéville castle museum	Lorraine
TBL 31	Saucer	End of 18th century	Embossed decoration, Lunéville castle museum	Lorraine
TBL 32	Bowl	End of 18th century	Embossed decoration, Lunéville castle museum	Lorraine
TBL 33	Creamer	End of 18th century	Embossed decoration, Lunéville castle museum	Lorraine
LNV 14	Plate	c. 1780	Third-firing polychrome decoration, tulip. Sey-Chazelles depot (Inv. No. 1101/15).	Lunéville
LNV 15	Dish with moulded rim	c. 1780	Third-firing polychrome decoration, rim with two flowers, Sey-Chazelles depot (Inv. No. 1101/18).	Lunéville

been destroyed by the fire, or thanks to the currently accepted stylistic analyses. TBL 14 is discussed in Calame (2009, 26–7) and TBL 15 in Choux (1979). In this study, two CaO-rich figurines (TBL 17, TBL 25) attributed to Cyfflé's Lunéville manufacture (1768–80) and analysed by Maggetti *et al.* (2010) will be included.

Sample preparation

From 24 ceramic objects, a small sample was obtained by cutting with a saw. Representative portions of these samples (3.6–15.7 g) were ground in a tungsten carbide mill after careful removal of the possibly contaminated surface. If available, small subsamples from broken objects were used for water adsorption and scanning electron microscopic analyses. Figurine TBL 14 was sampled at the base with a mini-saw, giving a small fragment (0.6 g), which was used for water adsorption and scanning electron microscopic analyses. Further, the amount of powder collected during the cutting of this object was too low for a chemical analysis by X-ray fluorescence, but enough for X-ray diffraction analysis.

Chemical analyses by X-ray fluorescence (XRF)

An amount of 2 g of powdered sample was calcined at 1000°C for 1 h to obtain the loss on ignition (LOI). Then, 0.700 g of calcined powder was carefully mixed with 6.650 g of MERCK spectromelt A10 ($\text{Li}_2\text{B}_4\text{O}_7$) and 0.350 g of MERCK lithium fluoride (LiF). This mixture was put into a platinum crucible and melted at 1150°C for 10 min (Philips® PERL X-2) in order to obtain a glassy tablet. These tablets were analysed for major, minor and trace elements using a Philips® PW 2400 wavelength-dispersive spectrometer (rhodium tube, 60 kV and 30 mA). Calibration was made on 40 international standards. The accuracy and precision were checked using laboratory reference samples. The error has been evaluated to be less than 5% for all elements analysed. The total of TBL 20 is slightly higher than 101 wt%. This is probably due to a too high measurement of the silica, because the same tablet, measured with the UNIQUANT method, gave 62.74 wt% SiO_2 , which is 1 wt% lower than the 63.72 wt% figure shown in Table 3.

Porosity

The water adsorption of whole fragments (0.6–9.6 g) of nine specimens was measured applying DIN 118132 (1995). The samples were dried for 24 h at a temperature of 100°C. After each measurement, the samples were allowed 24 h in the water and measured again until two successive measurements did not differ.

Mineralogical analyses by X-ray diffractometry (XRD)

The mineralogical composition was determined through powder X-ray diffraction, using the powder left over after the confection of the XRF tablets (Philips® PW 1800 diffractometer, $\text{Cu-K}\alpha$, 40 kV, 40 mA, 2θ 2–65°, measuring time 1 sec per step). No powder was available for TBL 3 and TBL 13.

Scanning electron microscopy (SEM)

Due to financial and sampling reasons, only a few samples could be analysed. Backscattered electron images (BSE) were collected with a scintillator-type detector out of polished samples,

Table 3 Bulk compositions of the specimens analysed by XRF (oxides and LOI in wt%, elements in ppm; $Fe_2O_3 = \text{total Fe as } Fe_2O_3$) and SEM-EDS (oxides in wt %; TBL 1-4, average of 10 area measurements $240 \times 150 \mu\text{m}$; TBL 2, 15 and 23, 400×300 , 190×200 and $300 \times 250 \mu\text{m}$, respectively)

An. no.	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	Ba	Cr	Cu	Nb	Ni	Pb	Rb	Sr	Y	Zn	Zr	Total	LOI
XRF																							
TBL 1	70.07	0.40	9.14	0.48	0.01	0.61	12.12	4.09	1.58	0.11	255	71	53	11	17	12 964	44	246	26	8	165	99.98	1.07
TBL 2	61.14	1.06	29.95	1.43	0.01	0.52	0.38	1.95	3.60	0.10	646	246	33	20	66	503	229	148	21	46	191	100.36	4.25
TBL 3	66.87	0.45	16.44	0.90	0.01	0.57	10.49	2.13	1.58	0.14	420	123	27	11	47	3 657	74	294	18	29	126	100.06	0.34
TBL 4	59.94	0.78	16.12	0.72	0.02	0.44	15.36	2.35	1.88	0.10	432	112	59	17	72	18 477	77	270	41	236	228	99.72	1.82
TBL 6	68.85	0.48	9.77	0.51	0.01	0.81	13.77	3.40	2.01	0.14	199	91	46	11	26	2 525	63	327	16	14	128	100.08	1.80
TBL 9	60.72	0.74	33.97	1.26	0.01	0.01	0.62	0.84	0.72	0.71	354	208	2	17	100	654	41	1156	65	73	228	99.89	0.41
TBL 10	69.06	0.45	9.64	0.45	0.02	0.55	14.06	3.39	1.26	0.26	200	59	17	13	19	4 832	120	285	17	5	156	99.75	4.90
TBL 11	76.75	0.63	8.61	0.90	0.01	0.39	9.69	1.02	1.07	0.41	266	23	5	13	8	3 084	77	326	27	13	187	99.88	3.76
TBL 12	76.77	0.62	8.64	0.90	0.01	0.39	9.72	0.97	1.07	0.41	291	13	9	15	8	3 062	75	324	27	12	186	99.91	5.24
TBL 13	66.95	0.61	14.39	0.82	0.02	0.36	13.64	1.39	0.61	0.10	108	61	46	14	17	11 840	9	380	45	90	182	100.19	1.89
TBL 15	61.06	0.70	15.75	0.60	0.01	0.36	16.43	0.34	1.73	0.06	291	123	35	17	62	27 880	11	295	57	297	256	99.98	0.56
TBL 16	57.82	0.78	19.33	0.82	0.01	0.40	17.03	1.14	1.70	0.05	276	104	18	17	117	16 970	87	283	38	305	183	100.93	0.50
TBL 18	63.29	0.92	31.49	1.18	0.01	0.12	0.65	0.94	0.75	0.35	261	157	11	23	76	2 401	52	642	67	153	227	100.11	0.45
TBL 19	59.22	0.78	16.72	0.67	0.01	0.38	16.16	1.03	1.95	0.06	249	92	116	17	91	25 234	71	262	47	554	220	99.68	0.58
TBL 20	63.72	0.81	15.39	0.81	0.01	0.37	15.61	1.03	1.68	0.06	244	90	36	17	31	17 023	70	341	42	157	240	101.36	1.98
TBL 21	61.74	0.72	15.45	0.79	0.01	0.46	14.56	1.39	1.74	0.06	226	88	24	16	32	24 843	62	314	49	104	247	99.53	2.68
TBL 22	61.81	0.83	16.60	0.76	0.02	0.40	15.56	1.13	1.69	0.06	281	108	16	18	29	11 681	78	289	34	72	210	100.16	0.55
TBL 23	57.15	0.77	19.23	0.80	0.01	0.40	16.99	1.12	1.81	0.06	283	92	19	18	113	16 832	91	283	38	243	184	100.18	2.18
TBL 30	61.97	0.74	33.83	1.17	0.01	0.03	0.72	0.83	0.76	0.51	302	188	10	17	96	1 799	34	858	50	79	216	100.96	0.42
TBL 31	61.68	0.74	33.88	1.17	0.01	0.02	0.75	0.84	0.73	0.51	319	174	7	17	96	861	35	870	49	75	212	100.64	0.33
TBL 32	61.30	0.75	34.02	1.29	0.01	0.03	0.57	0.85	0.61	0.51	322	180	12	18	104	956	38	925	53	106	217	100.27	0.29
TBL 33	59.60	0.78	16.84	0.70	0.02	0.42	16.81	0.99	1.87	0.07	299	88	31	19	100	22 731	72	307	45	665	222	100.56	0.58
LNV 14	68.78	0.54	10.57	0.53	0.01	0.62	13.99	2.72	1.55	0.19	240	57	18	12	20	9 343	67	310	26	11	158	100.54	7.84
LNV 15	68.47	0.52	10.99	0.56	0.01	0.60	14.56	2.32	1.50	0.17	237	60	19	12	21	8 665	65	275	23	12	149	100.67	8.04
SEM-EDS																							
TBL 2	60.9	1.0	29.2	1.3	1.6	0.4	1.3	3.5	0.8													100.0	
TBL 14	73.0	0.7	12.2	0.7	0.5	10.7	0.8	1.4														100.0	
TBL 15	61.3	0.6	17.0	0.6	0.3	15.8	0.3	1.5								26 000						100.0	
TBL 23	55.8	0.7	19.2	0.7	1.4	15.6	1.5	1.6								35 000						100.0	

Table 4 Water adsorption and modal compositions (in vol %)

An. no.	Water adsorption (%)	Quartz/flint/sand	Coarse CaO-rich area	Frit	Grog	Matrix	Pores	Total
TBL 3	9.5	23.5	3.2			63.8	9.5	100.0
TBL 14	13.5	26.6	0.6		2.4	56.9	13.5	100.0
TBL 15	13.2	12.9		5.3		70.0	11.8	100.0
TBL 18	12.6							
TBL 20	12.7	12.6	5.2	7.8		61.7	12.7	100.0
TBL 21	15.5	11.8	8.5	2.9		61.3	15.5	100.0
TBL 22	15.4	15.5	5.8	2.9		60.4	15.4	100.0
TBL 23	16.4	6.1	3.9	6.1		67.5	16.4	100.0
TBL 32	13.2							

using a Philips® FEI XL30 Sirion FEG electron scanning microscope. The samples were mounted in an epoxy block, flatly polished with a 0.5 µm diamond paste and then coated with a thin carbon layer. Chemical compositions were determined by energy-dispersive X-ray spectrometry (EDS), operated at a beam acceleration voltage at 20 kV and a beam current of 6.5 nA. Standardless quantification was performed using an EDAX-ZAF correction procedure of the intensities, using spot analyses (2 µm diameter) as well as larger area analyses of homogeneous areas. The detection limits for most elements were about 0.2 wt%. The reliability of the results was proven by measuring well-known glass and mineral standards (DLH2, Corning A-D and Plagioclase). The relative mean deviation for major and minor oxide components was 2% for concentrations in the range of 20–100 wt%, 4% for 5–20 wt%, 10–20% for 1–5 wt% and >50% for <1 wt%.

Quantitative phase (modal) analysis

Phase proportions of seven representative calcareous white earthenwares were determined by one digitized backscattered-electron (BSE) image analysis per sample, using the program Adobe Illustrator® (Patharea-Cs2-0-1.1b2.sit), integrating an area of $248 \times 185 \mu\text{m} = 85\,000$ points (Table 4). The siliceous white earthenwares were not integrated, due to the extreme difficulty of discerning the grog with the SEM.

Statistics

The statistical treatment of the chemical analyses was obtained using the program SPSS 13. Cluster analysis was performed with non-transformed data of 14 variables: SiO₂, TiO₂, Al₂O₃, Fe₂O₃, MgO, CaO, Na₂O, K₂O, Ba, Cr, Ni, Rb, Sr and Zr (average-linkage analysis, squared Euclidean distances, between groups).

RESULTS

XRF versus SEM-EDS

As the tiny TBL 14 fragment analysed could not be submitted to an XRF chemical analysis, but only to SEM-EDS, it became necessary to evaluate to what extent the two methods could be

compared. To that purpose, three samples (TBL 2, 15 and 23) were analysed by XRF as well as by SEM–EDS. As shown in Table 3, there are fortunately no great discrepancies between the chemical composition obtained by XRF and that by SEM–EDS, except in the case of magnesium and sodium.

Ceramic bodies

Bulk compositions The analysed sherds comprise two major compositional groupings: (1) CaO-rich—that is, calcareous—bodies; and (2) CaO-poor—that is, non-calcareous—aluminous–siliceous bodies (Table 3 and Fig. 3). The 19 CaO-rich samples can be divided further, according to their Al_2O_3 values, into two subgroups, with TBL 3 as outlier. This rough subdivision into two or more groups is also evidenced by other major oxide and trace element concentrations, such as SiO_2 , TiO_2 , Zr and Y (Fig. 4). All samples are characterized by very low iron and magnesium. Both CaO-rich figurines of Cyfflé (TBL 17, TBL 25; Maggetti *et al.* 2010) plot separately. TBL 11 and TBL 12 are chemically very similar—are they from the same object or from the same batch? The analyses of the six CaO-poor bodies show clear differences (Fig. 5). TBL 2 is low in Al_2O_3 , but high in TiO_2 , Fe_2O_3 , Na_2O , K_2O and Rb. TBL 9 has low SiO_2 and high Sr, while TBL 18 has high SiO_2 and high TiO_2 . The chemical similarity of TBL 30 and TBL 31 is most probably indicative of a derivation from the same batch.

Porosities The seven CaO-rich samples are very porous, as shown by the water adsorption (W.A.), which varies between 9.5% and 16.4% (Table 4). The two CaO-poor specimens have similar water adsorptions, of 12.6% and 13.2%.

Microstructures, modal analyses and phase compositions Almost all of the CaO-rich bodies are composed (see Fig. 6) of: (1) angular quartz with diameters not exceeding 340 μm —SEM–EDS

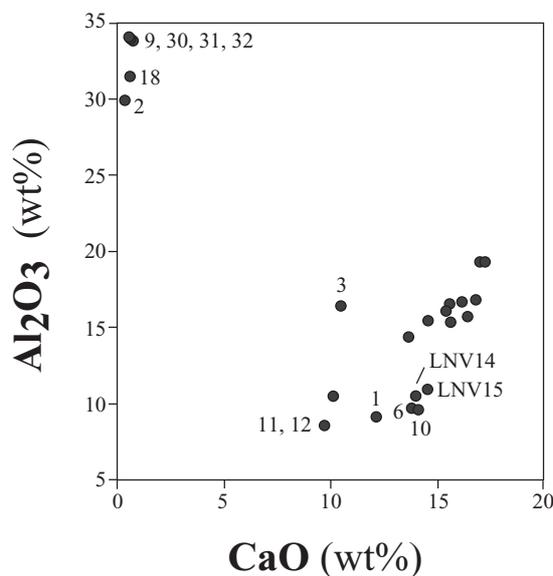


Figure 3 An Al_2O_3 –CaO correlation diagram.

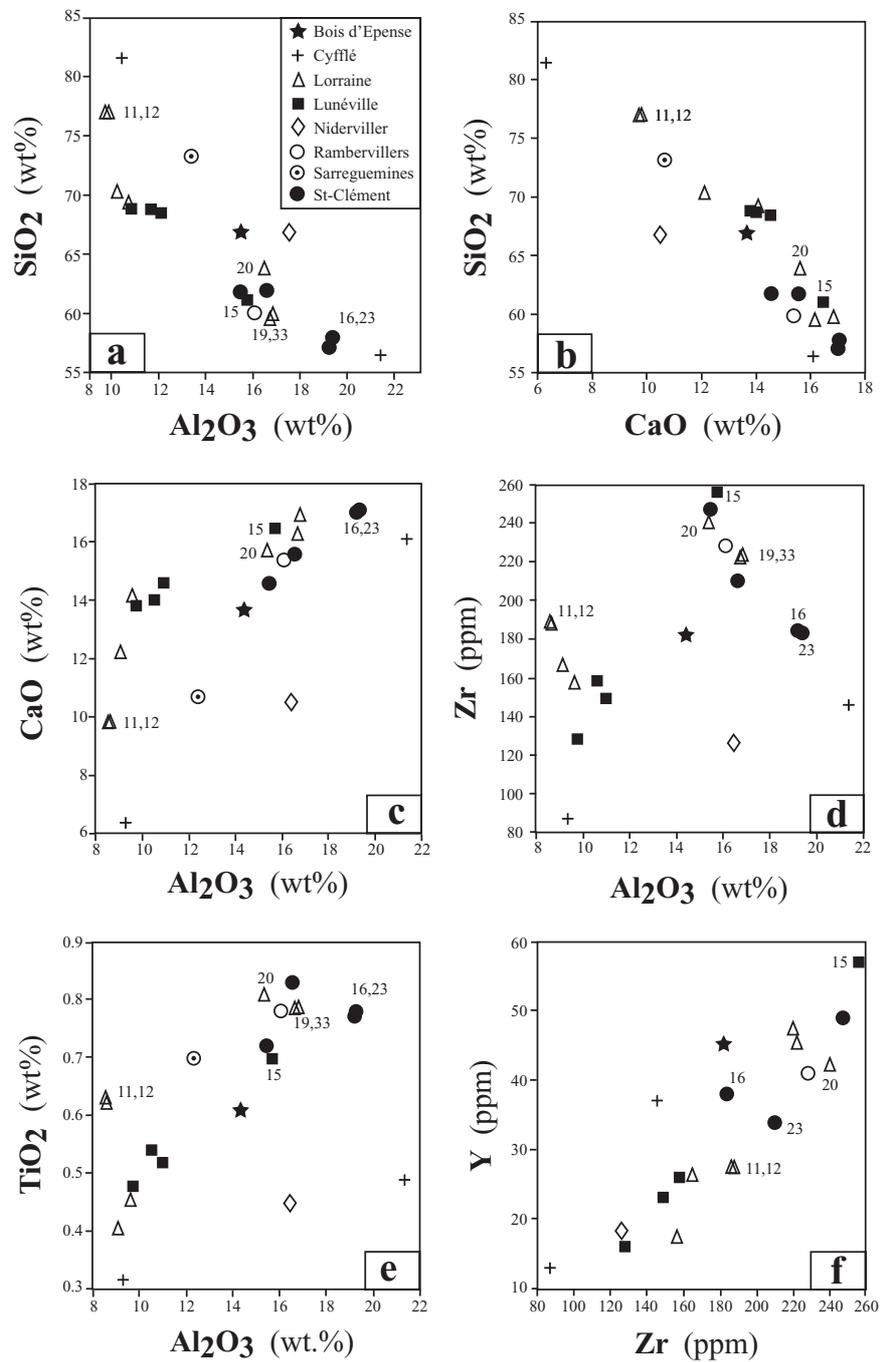


Figure 4 Body (bulk) compositions for the analysed CaO-rich samples displayed on bivariate plots of selected oxides and elements. Symbols describe the assumed provenance based on stylistic arguments (Table 2). Included are two Cyfflé's samples from Maggetti et al. (2010).

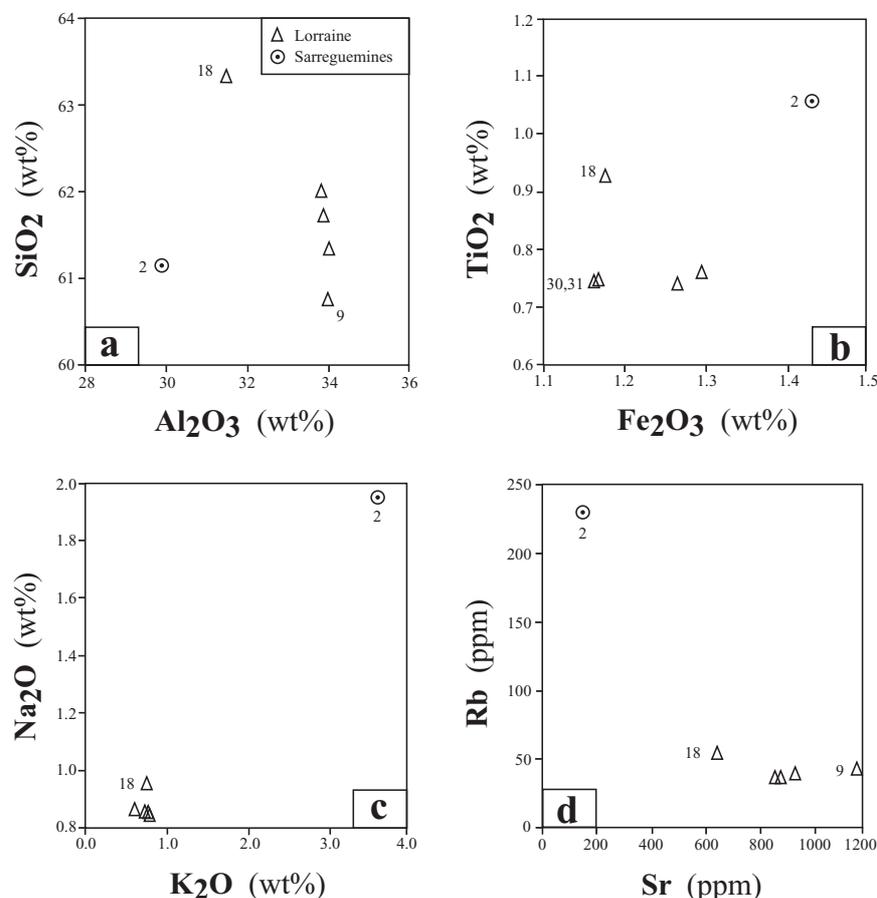


Figure 5 Body (bulk) compositions for the analysed CaO-poor samples displayed on bivariate plots of selected oxides and elements: for symbols, see Figure 4.

analyses of the brighter borders of these quartz particles indicate a fine layer rich in Si and Ca, where Ca silicates such as wollastonite have formed (Maniatis *et al.* 1983); (2) CaO-rich particles and more or less circular pores corresponding to primary particles rich in CaO—that is, carbonate or portlandite Ca(OH)₂—having reacted with the clay paste during the firing; (3) grog or Pb frits; and (4) a fine-grained matrix. The grog inclusions of TBL 14 are, as evidenced by Table 5, chemically highly variable, indicating their derivation from kaolinitic–illitic (high-Al), marly (high-Ca) or ‘normal’ (intermediate Al, K, Ca) clays. Almost all of the frits contain rounded quartzes and/or laths of a calcium silicate—probably wollastonite, according to the SEM–EDS point analyses—and of a second SiO₂ polymorph in the form of laths, which is probably cristobalite in view of its crystal habitus (Figs 6 (b) and (d)). The idiomorphic character indicates that cristobalite crystallized as a liquidus phase during cooling, and not during a subsolidus reaction such as devitrification. The rounded shapes of the quartzes are interpreted as having originated in a prograde fusion of primary quartz grains during the fritting process. Few glassy grains bear subidiomorphic tin oxide particles as inclusions (Table 5 and Fig. 6 (c)). To summarize, three frit types can be defined according to their (highly variable) chemical composition

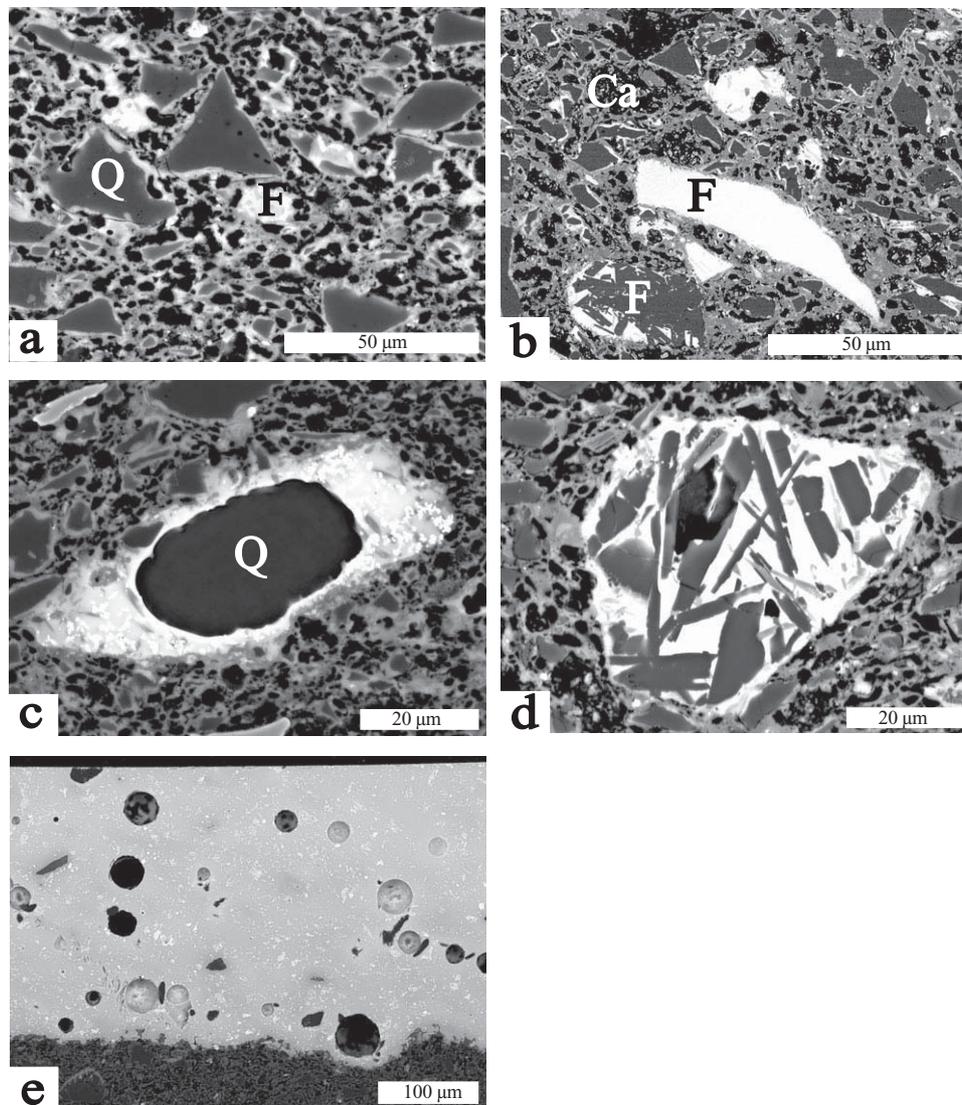


Figure 6 SEM backscattered electron images of CaO-rich bodies. (a) Sample TBL 15, showing angular fragments of quartz (Q) and frit (F), embedded in a fine-grained, porous matrix. (b) Sample TBL 20, showing rounded and angular fragments of lead-bearing frits (F), some with laths of a SiO₂ polymorph (black, cristobalite) and of wollastonite or Pb-feldspar (grey): Ca, CaO-rich areas. (c) A tin oxide (tiny white inclusions) bearing glass fragment with an irregularly shaped, corroded primary quartz (Q) in the core, implying resorption by the surrounding former melt (TBL 15). (d) A lead-glass fragment with laths of cristobalite (black) and Ca-silicate (grey)—TBL 21. (e) The typical glaze of a calcareous body opacified with cassiterites (small, white crystals) and with many gas bubbles: no reaction zone is visible between the glaze and the body—TBL 21.

(Fig. 7): (1) cassiterite-bearing lead alkali frit; (2) low-Ca lead frit; and (3) high-Ca lead frit. Frit 1 was found as rare inclusions in TBL 15 and TBL 21, frit 2 dominantly in TBL 20 and 21, and frit 3 mainly in TBL 15, 22 and 23. The compositions of frit 2 lie, as to be expected for mixtures made of quartz and lead oxide, on a SiO₂–PbO correlation line, whereas the high-Ca frits are

Table 5 Energy-dispersive X-ray spectrometry analyses of grog and lead-bearing glass fragments in CaO-rich bodies: spot and area (5×5 to $25 \times 35 \mu\text{m}$) measurements

An. no.	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	K ₂ O	SnO ₂	CaO	Fe ₂ O ₃	TiO ₂	PbO	Cl ₂ O	Total
TBL 14-16	1.1	1.8	9.2	69.1	6.5		10.4	1.2	0.7			100.0
TBL 14-17	0.7	1.8	8.3	70.6	2.1		15.4	0.8	0.3			100.0
TBL 14-18	1.0	0.4	33.1	48.1	10.1		4.6	1.7	1.0			100.0
TBL 14-19	2.0	1.0	33.1	52.4	7.8		0.6	2.5	0.6			100.0
TBL 14-20	0.8	1.3	1.9	82.7	1.6		8.7	1.7	0.3		1.0	100.0
TBL 14-26	1.0	0.2	1.5	74.9	5.9		3.6	12.7	0.2			100.0
TBL 15-1	0.4	0.4	6.5	53.0	2.7	10.5	4.2	0.6	0.3	21.3		100.0
TBL 15-15	0.3	0.3	8.6	67.5	4.2		8.7	0.6	0.4	9.2	0.2	100.0
TBL 15-22	0.4	0.8	10.5	56.7	3.6		16.6	0.9	0.9	9.2	0.3	100.0
TBL 15-25	0.5	0.4	19.2	56.4	5.6		6.3	0.7	1.1	9.6	0.2	100.0
TBL 15-29	0.5	0.4	10.8	62.1	4.9		10.2	0.5	0.4	10.1	0.1	100.0
TBL 20-1	0.8	0.6	3.8	63.5	4.5		2.1	0.9	0.2	23.4	0.2	100.0
TBL 20-2	0.9	0.9	4.7	56.4	5.8		2.7	1.2	0.4	26.8	0.2	100.0
TBL 20-3	0.8	0.5	6.3	58.8	6.4		4.8	0.5	1.2	20.4	0.3	100.0
TBL 20-4	1.4	0.5	8.5	57.0	4.6		3.2	1.0		23.6	0.2	100.0
TBL 20-5	0.4	0.5	2.8	81.3	2.4		3.2	0.5	0.2	8.6	0.1	100.0
TBL 20-6	0.4	1.0	3.1	69.8	3.2		10.3	0.6	0.3	11.2	0.1	100.0
TBL 20-7	0.7	0.8	4.1	68.9	4.2		6.0	1.1	0.2	13.9	0.1	100.0
TBL 20-8	0.4	0.9	3.8	72.3	3.5		6.7	0.8	0.2	11.3	0.1	100.0
TBL 20-9	0.3	0.4	3.7	75.6	3.7		2.6	0.5	0.2	12.8	0.2	100.0
TBL 20-10	0.7	0.8	5.8	60.7	5.9		5.9	0.7	0.2	19.1	0.2	100.0
TBL 20-11	0.6	0.6	6.7	62.5	4.9		4.7	0.8	0.3	18.8	0.1	100.0
TBL 20-12	0.8	0.6	3.8	57.8	5.4		2.5	0.9	0.3	27.8	0.1	100.0
TBL 20-13	0.8	0.6	4.6	58.2	5.9		4.4	1.2	0.4	23.7	0.2	100.0
TBL 21-4	2.5	2.1	4.8	46.6	1.8		2.0	0.7	0.4	38.3	0.9	100.0
TBL 21-11	1.2	0.2	7.3	54.9	3.7	8.7	2.8	0.5	0.1	20.4	0.2	100.0
TBL 21-12	0.8	0.4	4.3	64.1	2.3		2.8	0.8	0.4	23.9	0.2	100.0
TBL 21-13	1.0	0.7	5.0	73.4	2.7		4.8	0.8	0.3	11.2	0.1	100.0
TBL 21-14	0.9	1.2	3.8	67.0	2.2		7.6	0.9	0.5	15.7	0.2	100.0
TBL 21-15	0.8	0.3	4.7	74.9	2.1		7.0	0.9	0.2	8.8	0.3	100.0
TBL 21-16	1.6	0.6	6.2	59.5	3.7		5.8	1.0	0.4	21.0	0.2	100.0
TBL 21-17	1.4	0.9	6.2	58.6	3.7		5.1	0.9	0.4	22.5	0.3	100.0
TBL 21-18	0.4	0.7	2.0	83.2	1.0		3.7	0.4	0.6	7.8	0.2	100.0
TBL 21-19	1.1	0.8	13.9	54.8	3.4		18.0	1.1	0.4	6.3	0.2	100.0
TBL 21-20	0.8	0.5	5.1	35.9	2.2	36.1	5.4	0.6	0.2	13.0	0.2	100.0
TBL 22-11	0.7	0.5	8.2	72.1	2.5		11.1	0.8	0.4	3.6	0.1	100.0
TBL 22-12	1.1	0.6	10.6	67.1	5.0		7.8	0.6	0.3	6.8	0.1	100.0
TBL 22-13	1.4	0.4	11.3	66.7	5.9		3.5	0.8	0.6	9.2	0.2	100.0
TBL 22-14	0.6	0.6	6.6	67.7	2.5		16.3	0.7	0.6	4.3	0.1	100.0
TBL 22-15	1.2	0.7	12.9	62.7	5.8		8.8	1.0	0.4	6.4	0.1	100.0
TBL 22-16	1.1	0.5	17.5	55.6	3.9		14.9	1.0	1.0	4.3	0.2	100.0
TBL 22-17	1.1	0.3	15.2	59.0	7.3		9.1	1.3	0.5	6.0	0.2	100.0
TBL 22-18	1.0	1.2	29.0	44.3	1.4		14.0	4.7	0.6	3.7	0.1	100.0
TBL 22-20	1.0	0.9	9.0	63.8	4.1		12.6	1.3	0.6	6.5	0.2	100.0
TBL 22-22	1.1	0.7	13.9	58.3	4.4		15.2	0.9	0.6	4.7	0.2	100.0
TBL 23-11	0.9	0.5	6.3	66.3	3.0		11.3	1.1	1.7	8.6	0.3	100.0
TBL 23-12	0.9	0.2	11.0	61.5	5.3		12.3	0.6	0.2	7.9	0.1	100.0
TBL 23-13	1.3	0.7	8.7	60.1	4.9		9.3	1.0	0.2	13.6	0.2	100.0
TBL 23-14	1.4	0.7	6.3	67.3	3.6		5.4	1.2	0.2	13.6	0.3	100.0
TBL 23-15	0.8	0.6	5.9	71.8	2.8		9.5	0.8	0.5	7.1	0.2	100.0
TBL 23-16	1.4	1.6	6.2	61.0	3.7		10.7	1.7	0.5	12.9	0.3	100.0
TBL 23-17	0.6	0.3	12.7	65.7	3.3		11.8	0.7	1.4	3.3	0.2	100.0
TBL 23-18	1.6	0.7	7.1	59.1	4.7		3.1	2.3	0.5	20.5	0.4	100.0
TBL 23-20	0.8	0.4	17.7	52.5	4.5		17.6	0.7	1.1	4.6	0.1	100.0
TBL 23-21	1.1	0.6	17.9	55.2	7.5		12.3	0.7	0.4	4.2	0.1	100.0

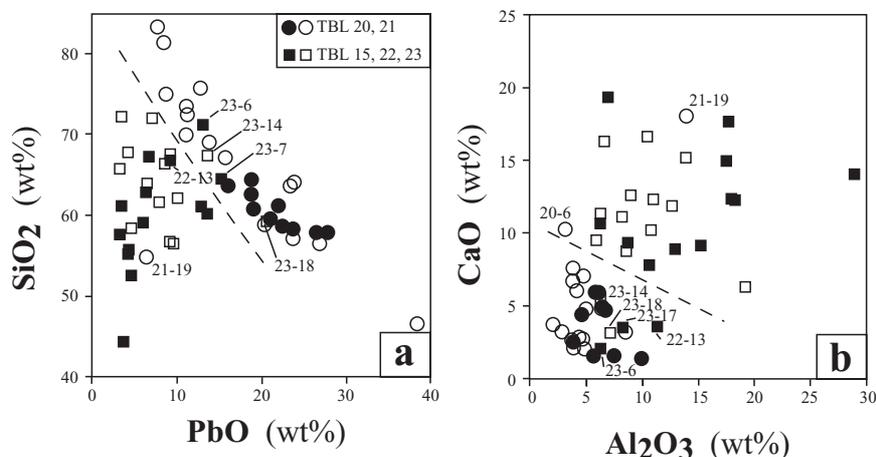


Figure 7 Bivariate plots of selected oxides of the lead frit temper in calcareous bodies. Solid symbols, only glass phase; open symbols, glass and crystals.

widely scattered. The matrices—that is, the former clay—of the seven analysed bodies are, as evidenced by Table 6, chemically similar, with high CaO and PbO (except TBL 14) between 0.5 and 3.2 wt%. Their overall chemical composition is close to that of kaolinitic–illitic clays. The modal proportions of the constituents, based on quantitative BSE-image analyses, are given in Table 4. The amount of former clay (matrix) shows less variation (57–70 vol%) than the quartz (6–27 vol%), the coarse CaO-rich area (0.6–8.5 vol%) and the frit (3–8 vol%). TBL 14 has 2.4 vol% of grog.

Three CaO-poor bodies (TBL 18, 30 and 32) contain, apart from very few grains of apatite, rutile and sphene, two major inclusion types (Fig. 8), embedded in a fine-grained, aluminous and siliceous matrix, made up of small platy grains of former clay minerals: (1) angular fragments of quartz with a maximum length of 340 μm (Fig. 8 (a)); and (2) angular grains of homogeneous, aluminous–siliceous inclusions (maximum length 205 μm), many with no apparent porosity (Figs 8 (b)–8 (d)). The fourth CaO-poor sample, TBL 2, shows only quartz as non-plastic inclusion. The second inclusion type fulfils two criteria of a grog temper: (1) an even distribution pattern in the studied samples; and (2) an internal fabric unrelated to the matrix fabric of the sample (Whitbread 1986; Cuomo di Caprio and Vaughan 1993). The chemical compositions of the grog grains are very similar for the three samples, with small intra-sample variation, and roughly resembling those of fired kaolinitic–illitic clays (Table 6). The matrices of the four analysed bodies are, apart TBL 2 with high K₂O, chemically similar. They differ from the grog compositions but also resemble roughly those of kaolinitic–illitic clays (Table 6).

Mineral associations The studied samples can be assigned, as shown by their X-ray diffractograms, to five different mineral associations:

- (a) Illite (110) + α -quartz + plagioclase + gehlenite + diopside + hematite + calcite (TBL 4, 15, 16, 19, 20, 21, 22, 23 and 33).
- (b) α -Quartz + plagioclase + gehlenite + diopside + hematite + calcite (TBL 6, 10, 11, 12, 14, LNV 14 and LNV 15).

Table 6 Energy-dispersive X-ray spectrometry analyses of matrix (without quartz and frit temper) in CaO-rich and CaO-poor bodies and of grog in CaO-poor bodies: averages of area (3×3 to $60 \times 40 \mu\text{m}$) measurements

An. no.	n	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	PbO	Cl ₂ O	Total
<i>Matrix of CaO-rich bodies</i>													
TBL 3	10	65.3	0.5	18.3	1.0	0.6	11.3	0.7	1.7		0.5	0.1	100.0
TBL 14	6	59.0	0.9	20.4	1.0	0.6	14.9	1.2	1.9	0.1			100.0
TBL 15	10	53.0	0.8	20.7	0.9	0.5	18.9	0.4	1.6		3.2	0.1	100.0
TBL 20	10	51.8	0.9	20.5	1.1	0.6	20.6	0.4	1.6		2.5	0.1	100.0
TBL 21	10	52.9	0.8	21.3	1.2	0.6	18.3	0.7	1.7		2.4	0.1	100.0
TBL 22	10	51.5	1.0	23.1	1.1	0.6	18.6	0.9	1.3		2.0	0.2	100.0
TBL 23	10	50.0	0.8	24.9	1.0	0.5	19.1	0.7	1.1		1.8		100.0
<i>Matrix of CaO-rich bodies without CaO, PbO and Cl₂O</i>													
TBL 3	10	74.2	0.6	20.7	1.1	0.7		0.8	1.9				100.0
TBL 14	6	69.6	1.1	24.0	1.1	0.6		1.4	2.2				100.0
TBL 15	10	68.2	1.0	26.6	1.1	0.6		0.4	2.1				100.0
TBL 20	10	67.4	1.2	26.7	1.4	0.8		0.5	2.0				100.0
TBL 21	10	66.8	1.0	26.9	1.5	0.8		0.9	2.1				100.0
TBL 22	10	64.7	1.3	29.1	1.5	0.7		1.1	1.6				100.0
TBL 23	10	63.2	1.0	31.6	1.3	0.6		0.9	1.4				100.0
<i>Matrix of CaO-poor bodies</i>													
TBL 2	5	60.0	1.1	32.6	1.3	0.8	0.5	0.3	3.5				100.0
TBL 18	5	63.4	0.9	32.6	1.0	0.4	0.6	0.3	0.7	0.2			100.0
TBL 30	5	60.8	0.6	34.8	1.1	0.3	0.7	0.3	0.9	0.4	0.2		100.0
TBL 32	5	59.8	0.8	36.0	1.2	0.4	0.6	0.1	0.6	0.4			100.0
<i>Grog fragments in CaO-poor bodies</i>													
TBL 18	11	51.2	0.1	42.6	1.1	0.4	1.1	0.6	2.8	0.1			100.0
TBL 30	14	49.1	0.1	44.9	1.0	0.4	1.3	0.3	2.5	0.3	0.1		100.0
TBL 32	10	51.6	0.1	42.7	1.3	0.5	1.0	0.2	2.1	0.5			100.0

- (c) α -Quartz + plagioclase + diopside + hematite + calcite (TBL 1).
- (d) α -Quartz + mullite + rutile (TBL 2).
- (e) α -Quartz + cristobalite + mullite \pm rutile (TBL 9, 18, 30, 31 and 32).

Associations (a)–(c) pertain to the CaO-rich, and associations (d) and (e) to the CaO-poor, samples. Calcite is a post-firing, secondary phase (Maggetti 1994) in the first three associations, as revealed by optical microscopy analysis. In association (e), both low- and high-temperature silica polymorphs are present. As shown by BSE imaging, the α -quartz occurs in these bodies as coarse, crushed and unreacted grains (Maggetti *et al.* 2010). No cristobalite or mullite were detected in the SEM analyses. Consequently, these phases are interpreted to occur as minute (sub- μm) crystallites.

Glazes

The glazes are not easy to study and to interpret; first, because the objects coming from the castle were probably chemically contaminated by the blaze of 2 January 2003—and, moreover, the extent of such a contamination cannot be evaluated because we have no means of comparing

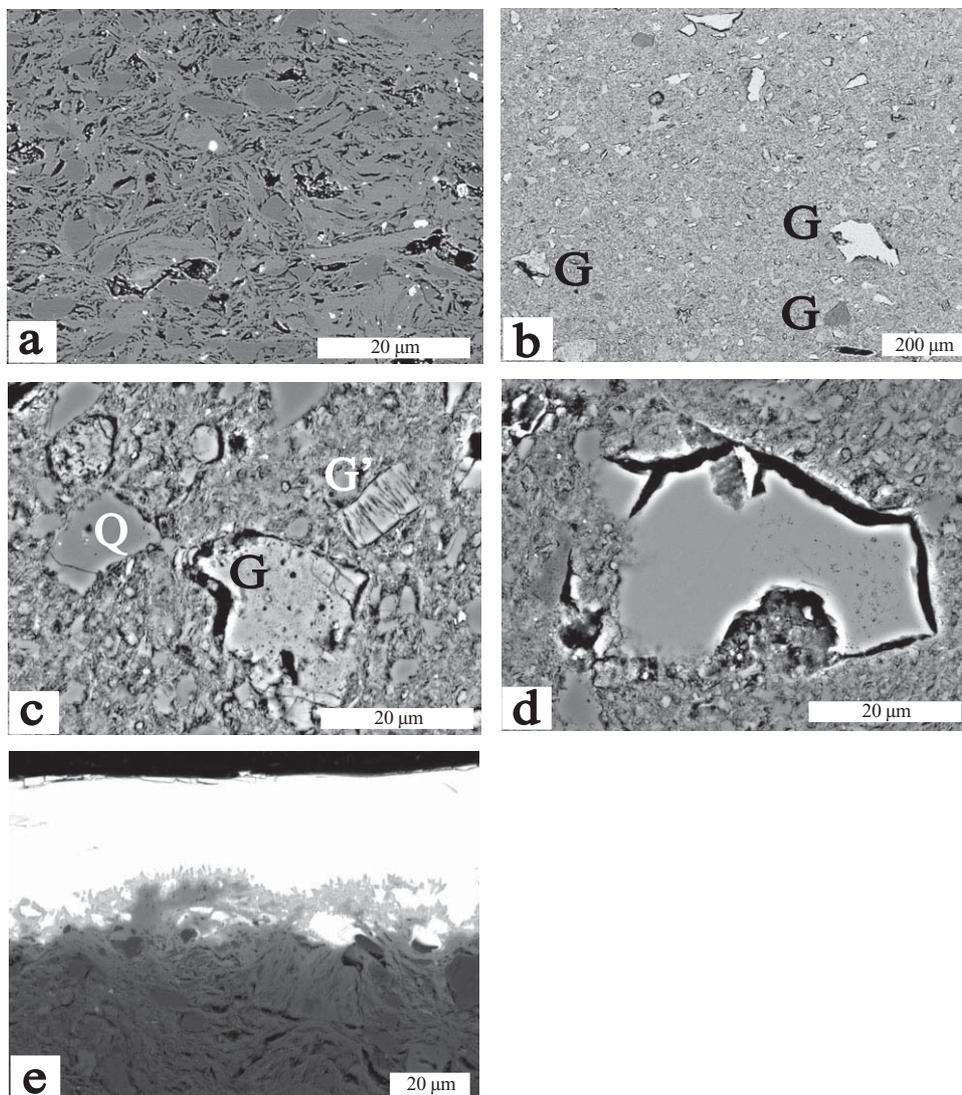


Figure 8 SEM backscattered electron images of CaO-poor bodies. (a) Quartz and coarse clay minerals (agglomerates = grog?)—TBL 2. (b) The body of TBL 18 with different grog (G) types. (c) Porous (G) and striated (G' = overfired kaolinite?) grog in TBL 30. (d) Grog with a homogeneous glassy (left) and a porous glassy (right) aspect, separated from the matrix by a crack—TBL 30. (e) The tiny body/glaze reaction zone in TBL 2.

them with identical uncontaminated objects—and, second, because it is impossible to obtain samples from intact pieces from museum or private collections.

White glazes These glazes, present on the CaO-rich, but not on the CaO-poor, bodies, are opacified by pigments of tin oxide white; that is, cassiterite (Fig. 6 (e)). SEM observations revealed the presence of few inclusions such as rounded quartz and K-feldspar as well as idimorphic laths of a SiO₂ polymorph, most probably cristobalite because of its shape. The

thickness varies from 100–140 μm (TBL 3) to about 300 μm (TBL 20–23) and 500 μm (TBL 15). Chemical compositions are reported in Table 7. The six analysed samples have similar oxide concentrations, with SnO_2 varying from 7 to 10 wt%. In zones where the cassiterite particles are agglomerated in clusters of 20–60 μm diameters, very high SnO_2 concentrations (24–51 wt%, TBL 20, $n = 11$) were measured. Point analyses of cassiterite-free areas showed mean SnO_2 values of 1.3 (TBL 20) to 1.8 (TBL 21) wt%. At the surface, no alteration/contamination layer was observed. Body/glaze interfaces show no interface due to chemical reactions, which indicates a double firing processes at moderate temperature.

Third firing decorations The chemical compositions of three coloured decorations are reported in Table 7. They show markedly differing SiO_2 and PbO concentrations, which rules out the possibility that these glazes, as well as the white glazes, were made using the same sand/lead mixture recipe. On the contrary, the green glaze shows some affinities with the transparent glazes of TBL 2 and TBL 18. The green glaze is characterized in marked CuO (1.8 wt%), and the violet decoration in MnO (0.4 wt%) and P_2O_5 (1.4 wt%). Cassius purple has the highest Na_2O content (9.2 wt%). The thickness is 25–30 μm for the green and the purple and about 20 μm for the violet layer.

Transparent glazes These glazes have irregular thicknesses, ranging from 25 μm (TBL 2), through 170–260 μm (TBL 18) and 2–50 μm (TBL 30), to 55–160 μm (TBL 32). Some glaze portions of TBL 18 are characterized by high ZnO values close to the surface (2.1 wt%), which decrease to 0.9 wt% close to the body (both area measurements). Area measurements of the glaze on TBL 30 revealed very low PbO throughout the whole glaze (<3 wt%), and irregular ZnO concentrations, varying from a minimum of 1.3 wt% to a maximum of 6.6 wt%. Apart from TBL 2, with a small Pb -feldspathic body/glaze interlayer of 18 μm (Fig. 8 (e)), no such body/glaze reaction zones could be observed in the other three samples, indicating that the glaze suspension was applied to a biscuit-fired body. TBL 2 differs in its chemical composition from TBL 18 and TBL 32, having no sodium but higher alumina (Table 7). Macroscopical examination showed that these transparent glazes are to be found on all non-calcareous, but also on some calcareous, bodies (TBL 1, 4, 6, 19 and 33), with the exception of the unglazed figurine TBL 14.

DISCUSSION

Chemical contamination

Burial contamination Such contamination is to be expected for TBL 9-12 and LNV 14-15, which were unearthed in excavations. Compared with the other specimens, their chemical composition is seemingly not affected by such processes, with the exception of phosphorus. TBL 9 has 0.71 wt% P_2O_5 , which is slightly higher than the maximum concentrations found in clays (Koritnig 1978). This higher value is interpreted as contamination through migrating P-rich solutions (Collomb and Maggetti 1996 and literature therein).

Contamination due to the blaze of 2 January 2003 The zinc values for many body samples exceed the maximum of 160 ppm of 454 clays and shales low in bituminous and carbonaceous matter from all over the world (Brehler and Wedepohl 1969). ZnO can be added to increase the whiteness of the body (Majewski and O'Brien 1987). But since concentrations higher than 250–300 ppm are only found in samples that suffered in the Lunéville castle blaze, they very

Table 7 Energy-dispersive X-ray spectrometry analyses of tin oxide opacified white glazes on CaO-rich bodies, third-firing colours on white opaque glazes and transparent glazes on CaO-poor bodies: averages of area (20 × 30 to 240 × 180 μm) measurements

	White glazes										Third firing colours on white glazes			
	TBL 3 n = 10 (min.-max.)	TBL 15 n = 10 (min.-max.)	TBL 20 n = 10 (min.-max.)	TBL 21 n = 7 (min.-max.)	TBL 22 n = 7 (min.-max.)	TBL 23 n = 5 (min.-max.)	TBL 23 n = 10 (min.-max.)	TBL 03 green	TBL 22 purple	TBL 23 violet				
Na ₂ O	1.8	1.6-1.9	2.2	1.8-3.4	2.6	2.3-3.6	1.6	1.4-1.8	1.2	1.1-1.3	1.4	9.2	2.4	
MgO	0.2	0.1-0.3	0.1	0-0.3	0.2	0.2-0.3	0.3	0.2-0.4	0.2	0.1-0.3	0.2	2.4	2.2	
Al ₂ O ₃	4.7	4.5-4.9	5.0	4.8-5.6	4.9	4.7-5.1	5.9	5.7-6.1	4.2	4.1-4.3	2.5	2.6	2.3	
SiO ₂	48.0	47.1-49.1	45.3	43.3-46.8	45.9	44.2-47.3	47.6	47.6-48.3	45.2	44.4-46.1	30.5	45.9	57.9	
K ₂ O	2.3	2.2-2.4	3.2	2.6-3.5	2.6	2.3-2.8	2.9	2.8-3.0	2.7	2.3-2.8	0.9	3.0	2.4	
SnO ₂	8.2	7.5-8.6	7.2	6.3-7.8	9.3	8.3-10.9	9.8	9.0-10.4	10.0	9.7-10.4	0.5	7.4	5.4	
CaO	1.0	0.9-1.0	0.4	0.3-0.6	0.4	0.4-0.6	1.0	0.9-1.2	0.7	0.5-0.9	0.7	5.4	5.4	
Fe ₂ O ₃	0.6	0.5-0.6	0.1	0.4-0.5	0.4	0.3-0.5	0.6	0.5-0.6	0.5	0.4-0.5	0.6	0.7	1.2	
TiO ₂														
PbO	32.4	31.5-33.7	35.3	34.4-35.9	32.9	32.5-33.9	29.9	29.3-30.9	34.4	33.5-35.1	60.4	22.3	23.9	
CuO											1.8			
MnO													0.4	
P ₂ O ₅													1.4	
Cl ₂ O	1.0	0.8-1.2	0.9	0.1-1.2	0.7	0.1-1.3	0.5	0.2-0.6	0.8	0.6-1.0	0.5	1.1	0.5	
Total	100.0		100.0		100.0		100.0		100.0		100.0	100.0	100.0	

	Transparent glazes			
	TBL 2 n = 3 (min.-max.)	TBL 18 n = 6 (min.-max.)	TBL 32 n = 4 (min.-max.)	
Na ₂ O				1.3-1.6
MgO	0.2	2.4	1.5	0.2-0.3
Al ₂ O ₃	7.8	0.1	0.3	3.4-3.7
SiO ₂	30.5	3.4	3.5	37.2-40.7
K ₂ O	0.6	32.6	39.1	2.1-2.3
CaO	0.1	1.1	2.2	2.1-2.4
Fe ₂ O ₃	0.4	1.4	2.3	0.5-0.8
TiO ₂	0.2	0.5	0.6	
PbO	60.2	57.3	50.2	48.7-52.1
ZnO		0.9	0.3	
P ₂ O ₅		0.1	0.3	
Cl ₂ O		0.1	0.4	
Total	100.0	100.0	100.0	0.2-0.5

probably reflect chemical contamination by zinc vapours, coming out of melting metallic objects, during the blaze (Maggetti *et al.* 2010). The decreasing ZnO concentration in the lead glaze of TBL 18, from the surface to the boundary with the body, is also interpreted to originate from this blaze. This is supported by TBL 30, which shows a severe alteration of the original lead-rich glaze to PbO values below 3 wt% and ZnO concentrations up to 6.6 wt%. Both samples have low Zn in their ceramic bodies (TBL 18, 153 ppm; TBL 30, 79 ppm), which apparently contradict such contamination processes. However, profile measurements of TBL 30 showed significant ZnO concentrations in the outermost 1000 µm of the ceramic body (1.25 wt% ZnO at a distance of 100 µm to the glaze, 1.7–1.8 wt% at 200–400 µm, 1.4 at 600 µm, 0.5 at 800 µm and 0.0 at 1000 µm).

Classification

All studied objects have porosities higher than 2% and therefore belong to the group of ‘white earthenware’ (Fig. 2). The CaO-rich bodies pertain to the group of the ‘calcareous white earthenware’ and the CaO-poor bodies to ‘siliceous white earthenware’, in terms of modern classification. However, TBL 2 could also be classified as ‘feldspathic white earthenware’ (see below).

Provenance

CaO-rich bodies Figure 4 has shown the complexity of the composition of the studied ‘calcareous white earthenware’ specimens, indicating clearly that different recipes were used. The question is: one centre with different recipes or different centres with differing recipes? As yet, no white earthenware manufacture in Lorraine has been excavated, making it impossible to safely establish groups of chemical references with local materials, as in the case of ‘overfired’ goods, for example. We must therefore base our conclusions on the historical and stylistic arguments of Table 2, which attribute more than half of the objects studied to a very specific manufacture in Lorraine. The dendrogram obtained by cluster analysis shows different groups (Fig. 9). TBL 14 was not taken into consideration, since we only have the major elements; but Figure 4 clearly shows that it differs chemically from the other objects, which would indicate that the Sarreguemines manufacture used another recipe. The interpretation of the dendrogram is based on the hypothesis that the manufactures had exchanged neither recipes, nor paste nor biscuits. In order to place the cut in the dendrogram, we let ourselves be guided primarily by stylistic factors. It was inserted in the place of greatest convergence between stylistic approach and chemical composition, at a distance of 6.7 (the broken line in Fig. 9), making it possible to see seven groups. The first contains nine samples, four of which are attributed stylistically to the Saint-Clément manufacture. We would then be justified in thinking that the five other pieces could also have been produced in that workshop. The attribution based on style for TBL 4 to Ramberviller and for TBL 15 to Lunéville would then have to be reconsidered. TBL 17, a biscuit by Cyfflé, makes up the second group, which is not surprising if it comes from his workshop. The third group contains five pieces, three of which are attributed to the Lunéville manufacture, on the basis of style considerations. TBL 1 and TBL 10, of unknown provenance, would thus also originate from Lunéville. The fourth group includes sample TBL 3, attributed to Niderviller, and is different from the others in that it does not have a frit as temper. TBL 11 and TBL 12, of unknown provenance, form the fifth group. Through lack of reference to objects whose attributions has been more or less established, the origin of those two objects cannot be specified. TBL 13, attributed stylistically to the Bois d’Épense manufacture, is the only sample in the sixth group.

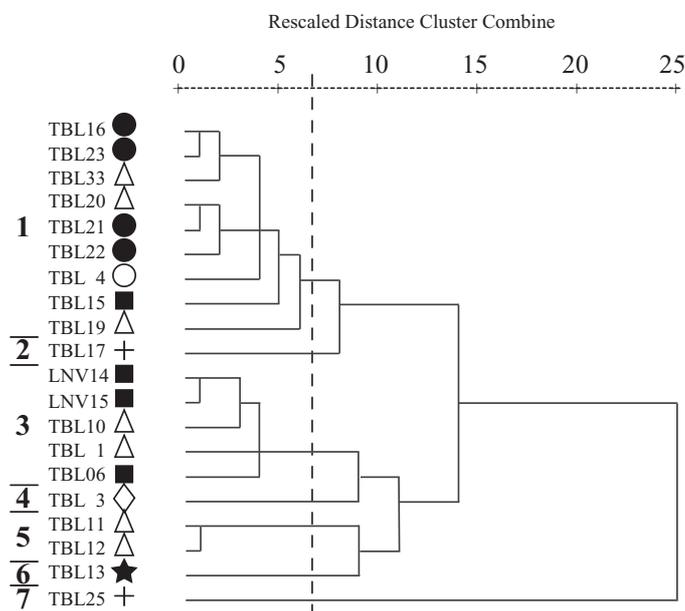


Figure 9 Grouping of the CaO-rich specimens by cluster analysis. The biscuit figurine TBL 14 was not included, because no trace elements were measured. The dashed line represents the cutting line of the tree, used for the definition of seven groups. Symbols are as in Figure 4.

The chemical analysis confirms its alleged origin. Figurine TBL 25, attributed to Cyfflé (Maggetti *et al.* 2010), makes up the seventh group. It is the most distant of all pieces because its SiO₂ content (81.55 wt%), for example, is the highest.

The factors on which this classification is based can be deduced from a thorough analysis of the chemical compositions (Table 3) and on the position of each sample in the binary diagrams (Fig. 4). It follows that the analysis of the dendrogram corroborates the stylistic analysis and makes it possible, with all due caution, to tentatively attribute the ‘Lorraine’ pieces of Table 2, with the exception of TBL 11 and 12, to the workshops of Lunéville or Saint-Clément. The two pieces by Cyfflé (TBL 17 and TBL 25) chemically stand apart and so cannot be attributed to either of these manufactures. Consequently, if they were really produced in Cyfflé’s manufacture, we must consider the possibility that Cyfflé followed two different recipes to make them. If that were not the case, we would have to consider a provenance from a manufacture not yet analysed. But for the time being, so long as we have no new elements, their origin in the Cyfflé manufacture is not in doubt. As a conclusion to this stylistico-chemical line of argument, the ‘calcareous white ware’ analysed can be grouped as follows:

Bois d’Épense	TBL 13
Cyfflé	TBL 17, 25
Lunéville	LNV 14, LNV 15, TBL 1, 6, 10
Niderviller	TBL 3
Saint-Clément	TBL 4, 15, 16, 19, 20, 21, 22, 23, 33
Sarreguemines	TBL 14
Unknown	TBL 11, 12

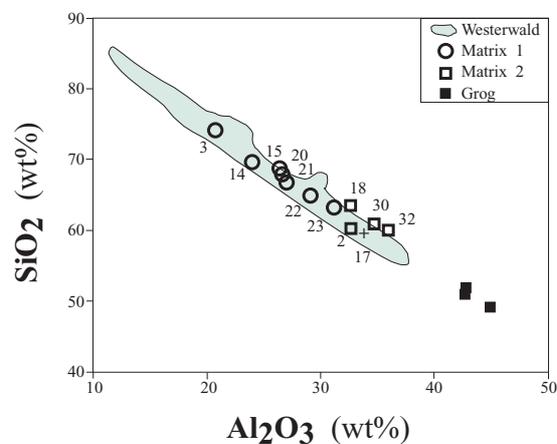


Figure 10 An $\text{SiO}_2\text{-Al}_2\text{O}_3$ diagram illustrating the range of 68 white-firing kaolinitic-illitic clays from the Westerwald (Schüller 1974). The matrices of CaO-rich (Matrix 1) and CaO-poor (Matrix 2) bodies fall in the field of the Westerwald clays. Grog from CaO-poor bodies plots outside this field, but on the same correlation line.

The chemical compositions of the Lunéville (L) and the Saint-Clément (S) productions are, as shown by three major oxides, different and their ranges astonishingly small (in wt%): SiO_2 (L 68–70, S 57–64), Al_2O_3 (L 9–11, S 15–20) and CaO (L 1–14, S 15–17).

CaO-poor bodies Figure 5 shows a very clear difference in composition for TBL 2 (Sarreguemines) and TBL 18 (provenance unknown, but from Lorraine) when compared with the other pieces analysed. Therefore, the Sarreguemines workshop used a different mixture. Unfortunately, in order to delve deeper, we would have to have at our disposal a greater number of objects analysed, better-documented stylistic attributions and established chemical reference groups.

Recipes

Ceramic bodies The microstructural analysis of seven CaO-rich bodies shows clearly that we are dealing with an artificial body, made from a white-firing clay, to which were added calcined and ground flint or sand, frits and chalk. In fact, the angular edge of the quartz inclusions—that is, the former flint or sand—is evident proof that these grains were added to the paste after grinding. The mineralogical nature of this clay paste can be deduced from the chemical composition of the matrices of the analysed specimens (Table 6). Chemically, they concur perfectly with 68 white-firing kaolinitic-illitic clays from the Westerwald (Fig. 10). The $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratios of the pieces analysed vary greatly, indicating most probably different clay pits or different clay layers in the same extraction site. At present, it is impossible to say for sure, but the correlation of the position of the pieces analysed (Fig. 10) to their provenance (Table 2) would tend to indicate that the manufactures used different clay pits or had different providers. Six of the seven pieces analysed by SEM-EDS, all of them dated to the end of the 18th century, do not contain any grog, while the quail TBL 14, an early 19th century sample, does. This is perfectly in keeping with the observation that grog seems to be an element found in recipes only from the late 18th or the early 19th century onwards (Table 1). Chemically, the frits of the analysed samples are not lead-free alkaline frits, but lead-bearing to lead-rich frits (Tite *et al.* 1998),

which explains the high lead content of the ceramic bodies. According to Table 1, this presence of Pb frit debris would place the pieces in the 19th century rather in the 18th. But their style and decoration are totally 18th century, which leads us to believe that the recipes in Table 1 do not reflect the enormous variety in recipes of the 18th century and that Pb frits may have been used long before the 19th century. These frit fragments, as shown by the SEM pictures, apparently survived the firing as separate particles with not much effect as a flux. On the other hand, the possibility cannot be excluded that some of the matrix lead could also derive from very finely powdered glass particles. If we accept the hypothesis that TBL 15, 20, 21, 22 and 23 come from the Saint-Clément manufacture (Fig. 9), it follows that several recipes were obviously used in this particular location. One used cassiterite frits—that is, those prepared for the lead glazing typical of faïence (let us recall that white ware and faïence ware were commonly produced in the same manufacture), while another used almost exclusively low-Ca and a third mostly high-Ca frits. The recipe for a pore-free mixture can be calculated by: (1) transforming the modal analysis into a porosity-free *Modus*; (2) converting all the CaO from the bulk analysis into wt% Ca carbonate (calcite, CaCO_3); and (3) converting modal quartz, frit, grog and matrix from vol% to wt% using ideal or inferred (3.0 for the Pb frit) specific weights. The results in Table 1 cannot be compared straightforwardly with the old recipes, because they refer to a porosity-free mass—unlike the old recipes, which call for porous clays. But they do make it possible to realize that there was no one single recipe in Saint-Clément, since the recipe varies for the five pieces attributed to that manufacture (TBL 15, 20, 21, 22 and 23). Indeed, the flint and frit contents vary significantly, with a rather constant calcite (= chalk) admixture. These objects also stand out quite clearly from the TBL 3 (Niderviller) and TBL 14 (Sarreguemines) recipes, thus providing proof that these two manufactures used other types of mixtures. It is surprising to note such a variety of recipes within the same manufacture as well as between manufactures, with pastes consisting of artificial mixtures of very similar raw materials (chalk, clay, flint, frit and sand). We must conclude that the manufactures constantly adapted their recipes in order to improve on the mechanical quality of their products.

The six CaO-poor bodies are an artificial mixture of crushed flint or sand, grog and kaolinitic–illitic clay, as evidenced by the SEM images (Fig. 8). The unusually high Sr values of almost all of them are puzzling (Fig. 5 (d)). They cannot be related to a specific admixture. The use of soda as a flux (Boch, cited by Peiffer 2007, 94) would explain the high Na_2O content of TBL 2, but this oxide is correlated with K_2O and Rb, which could be more indicative of a feldspathic flux. Therefore, TBL 2 could also be classified as ‘feldspathic white earthenware’. The kaolinitic–illitic clays (matrices) of these objects have lower SiO_2 contents, but higher Al_2O_3 contents, than those used for the CaO-rich bodies (Fig. 10). Their grog has even higher alumina values and plots in the same figure on the correlation line, an argument for a derivation from high-fired kaolinitic–illitic clays, most probably from the Westerwald region too.

Glazes One can wonder why the potters used white glazes—that is, traditional faïence glazes—in which the colour of the body was hidden by the opacity of this glaze to cover a biscuit, which showed white after the first firing. The use of such an expensive material as tin does not seem at all obvious at first sight, but according to Brongniart (1844), this procedure could have been motivated by the fact that the ‘siliceous white earthenware’ glaze was soft, easily altered and subject to crazing. The use of a hard tin-glaze, well adapted to a CaO-rich body such as that of traditional faïence and ‘calcareous white earthenware’—the thermal expansion coefficient of both bodies matches those of lead-alkali glazes (Tite *et al.* 1998; Tite 2009)—made it possible to avoid these drawbacks.

The chemical variation for the six white glazes is low (Table 7), indicating that the potters applied more or less identical, well-controlled recipes.

The green glaze can be classified as a high-lead glaze, and the purple and violet as lead-alkali glazes (Tite *et al.* 1998).

The three transparent glazes pertain to the high-lead type containing 50–60 wt% PbO (Tite *et al.* 1998). The variation of PbO is large and, as to be expected, correlated with SiO₂ (Table 7). Seemingly, different recipes were used.

Firing conditions

‘Calcareous white earthenware’ The microstructure of these samples can be classified as ‘extensive vitrification’, which develops at firing temperatures of about 1000°C (Maniatis and Tite 1981; Kilikoglou 1994; Wolf 2002). The ancient firing temperatures can also be estimated by comparison of their phase associations (a)–(c) with the phase evolution in experimentally fired raw materials with a similar calcareous composition (Maggetti 1982). For association (a), these inferred temperatures were most probably in the range 800–900°C, and for (b) and (c) in the range 900–1050°C. Such temperatures fall in the usual range of the French faience kilns of the 18th century (Rosen 1995; Maggetti 2007).

‘Siliceous white earthenware’ Their XRD phase associations (c) and (d) are characterized by high-temperature crystals such as mullite and cristobalite. As shown by experimental firings of refractory, Fe-poor clays (Maggetti and Rossmann 1981; Maggetti 1982), mullite forms at temperatures over 950°C and cristobalite at temperatures higher than 1050°C. Accordingly, TBL 2 was most probably fired in the range 950–1050°C, and the others at temperatures above 1050°C. Therefore, TBL 2 may have been fired in a standard faience kiln, but not the others, since their inferred ancient firing temperature is higher than the usual temperatures for this type of kiln. They could well have been fired in a bottle kiln, in which temperatures around 1200°C were common. These high temperatures could also have been attained in the Lunéville castle fire, but then it would be necessary to explain why the fire only affected the ‘siliceous’ and not the ‘calcareous white earthenware’.

CONCLUSIONS

In the late 18th and early 19th centuries, the Lorraine manufactures produced two types of ‘white earthenware’, calcareous and siliceous. They are artificial pastes, obtained by mixing an imported refractory white-firing clay with temper (calcined and crushed flint or sand, crushed biscuit) and flux (chalk, frit) for the CaO-rich bodies, and without flux for the CaO-poor bodies. The recipes and the techniques, which can be derived from archaeometric analyses, agree with the manuscripts of the late 18th century, and the technical treatises of the early 19th century. The raw materials were indeed the same, but each manufacture used its own recipe(s), different from the others. Consequently, the productions from each manufacture can be recognized by their chemical composition; which, through chemical analyses, enables the attribution of objects to the production centres—in particular, to the two main centres of Lunéville and Saint-Clément—and to confirm or reject attributions based on purely stylistic arguments.

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