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Neutron activation analysis of the Terra Sigillata from La Péniche

by *M. Maggetti**, *M.F. Ferreira Marques*** and *P.A. Schubiger***

Abstract

50 Terra Sigillata sherds from the pottery workshop of La Péniche were studied by NAA. The results confirmed the previous analyses by XRF that both Terra Sigillata groups of La Péniche are chemically homogeneous and quiet similar. Two sherds are of probable foreign origin. The comparison with the known analyzed european Terra Sigillata manufactures show the possible discrimination of the La Péniche production by some element pairs (e.g. Sc/Cs or La/Cr). For Ba, K_2O, MnO, Cr, Rb and Fe_2O_3 the NAA yields slightly higher values than the XRFA.

1. Introduction

The Terra Sigillata from the pottery workshop of La Péniche (Vidy/Lausanne) was studied detailed by mineralogical and porosimetrical methods. The major element and trace element chemistry was worked out by XRF analysis. The results of all these investigations were published in several papers (MAGGETTI and KUEPFER 1978, KUEPFER and MAGGETTI 1978, MAGGETTI 1980a). The paper of MAGGETTI (1980b) compares the Terra Sigillata with the common pottery of the roman vicus Lousonna while HEIMANN and MAGGETTI (1980) and MAGGETTI and HEIMANN (1979) are focused on the gehlenite problem. The trace element analyses were completed with the help of the neutron activation method which results are presented and discussed in this paper.

2. Experimental

50 sherds were analyzed. From each one 150 to 250 mg was taken, powdered and filled into ultrapure polyethylene containers. 6 sample containers and 3 standards were irradiated together in one batch. Standards were five powdered ceramics of the Centre de Recherches Archéologiques Médiévales, Caen (LAHA-

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NIER et al., 1972, PIGEAT et al., 1974). However new values of elemental concentrations were determined against the PERLMAN/ASARO's standard as described earlier (FERREIRA and SCHUBIGER 1980). Each batch was irradiated twice as follows:

1) Irradiation for 3 min by a flux of $1.3 \cdot 10^{13} n \cdot cm^{-2} \cdot s^{-1}$, cooling time of about 20 h and a counting time of 1 h.
2) Irradiation with $1 \cdot 10^{17}$ n·v·t and after a longer cooling period (7 to 11 d) a measure of 3 h.

The counting place has a 40 cm^3 Ge(Li)-detector and a ND 6620-multichannel system. For the actual conditions the resolution of the detector was 2.7 keV for the 1332 keV γ-line of ^{60}Co. The stored data have been evaluated by the γ-spectra evaluation program JANE (SCHUBIGER et al., 1978). The following nuclides have been used for the determination of the elements.

First counting: ^{152m}Eu (T = 9.3 h, 841 keV γ-line), ^{72}Ga (40.1 h, 834 keV), ^{56}Mn (2.56 h, 846 keV), ^{42}K (12.36 h, 1525 keV), ^{140}La (40.2 h, 1596 keV), ^{24}Na (15 h, 1368 keV).

Second counting: ^{131}Ba (11.5 h, 496 keV), ^{60}Co (5.27 a, 1332 keV), ^{51}Cr (27.7 d, 320 keV), ^{134}Cs (2.05 a, 796 keV), ^{152}Eu (12.4 a, 1408 keV), ^{140}La (40.2 h, 1596 keV), ^{177}Lu (6.71 d, 208 keV), ^{147}Nd (10.98 d, 531 keV), ^{86}Rb (18.7 d, 1077 keV), ^{46}Sc (84 d, 889 keV), ^{182}Ta (115 d, 1221 keV), ^{160}Tb (72.1 d, 966 keV), ^{175}Yb (4.2 d, 397 keV), ^{95}Zr (64 d, 856 keV).

3. Discussion

With respect to the adherance of the glossy clay layer, the Terra Sigillata from La Péniche can be divided in two groups:

a) Terra Sigillata of good quality (TS, with firmly adhering glossy clay layer)
b) Terra Sigillata of worse quality (TH, with badly adhering glossy clay layer).

We tested 25 sherd from each group previously analysed by XRFA (KUEPFER and MAGGETTI 1978, MAGGETTI 1980a).

3.1. COMPARISON OF BOTH TERRA SIGILLATA GROUPS

The XRF analysis show that both Terra Sigillata groups are very similar in its chemical composition. This is also true for the trace element chemistry determined by NAA (table 1, fig. 1). Slight differences arise only for some elements as Na_2O, K_2O, Rb, Cs and Ba. Cluster analysis corroborated the close grouping of the analyzed 50 sherds. At the 96% confidence level and by calculating with 17 analysed elements (without Zr) only six sherds (no 20, 44, 58, 65, 66, 95) do not belong to the sherd population.

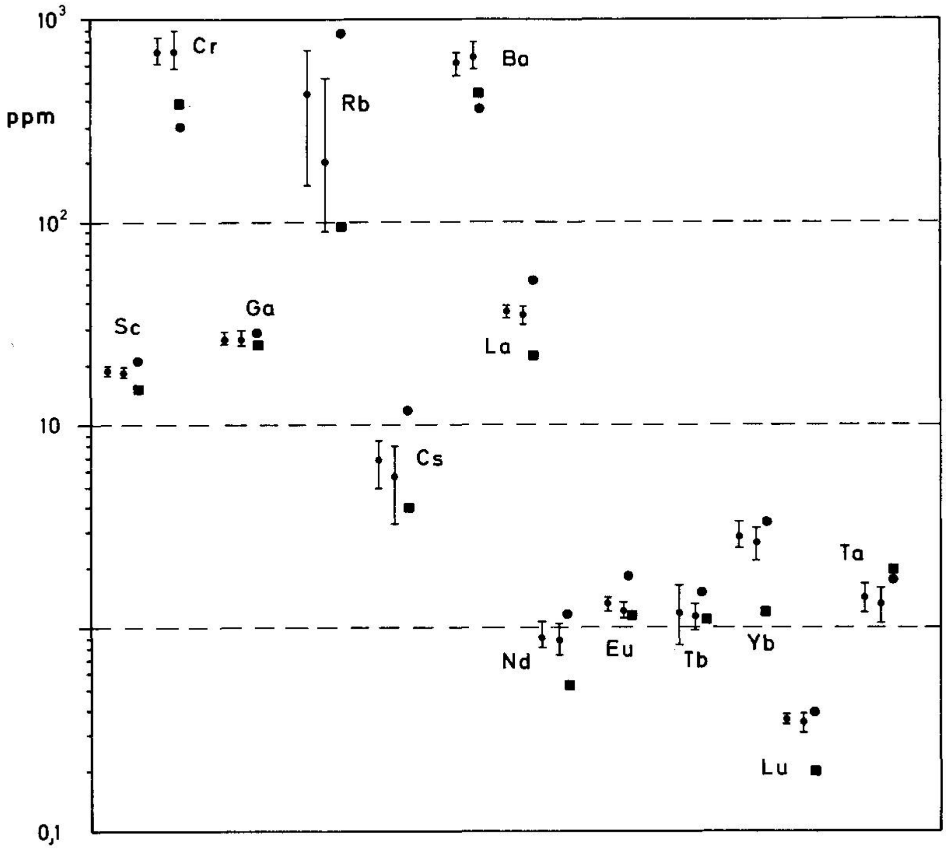

Fig. 1 Trace element content of the Terra Sigillata as determined by NAA. Mean values and standard deviations for the good quality (TS, left) and the worse quality (TH, right). Dot = sherd no 20, square = sherd no 95.

MAGGETTI (1980a) presented arguments for the location of the sherd no 20 to the pottery centre of La Graufesenque. This is now further confirmed by the concentrations of Ba, La, Eu, Cr, Na_2O and Fe_2O_3 which are significantly higher or lower than the reference group of La Péniche (fig. 1) and which plot close to the group of La Graufesenque (fig. 2, 3).

The sherd no 44 has only a lower content of Cr. The specimens no 65, 66 have significantly lower Cr and higher K_2O-contents, while no 58 has lower Rb and K_2O. KUEPFER and MAGGETTI (1978) argued that the high K_2O-content of no 65 and 66 was a sufficient argument for a foreign origin of these two sherds. However, the additional informations by NAA cannot support this thesis, as these specimens belong for 16 elements to the La Péniche reference group – two elements are not sufficient for discriminating them with a high statistical probability. It is therefore assumed that no 44, 58, 65 and 66 were made locally from a somewhat different clay.

Finally no 95 differs markedly in its La, Lu, Nd, Yb and Fe_2O_3-contents which are significantly lower than the La Péniche group (fig. 1). This could perhaps belong to an imported object, but no sure correlation can be established with an actually analysed reference group (fig. 2, 3). It is noteworthy that this

Table 1. Neutron activation analysis of the Terra Sigillata from La Péniche.

	TS (n = 23)			TH (n = 25)			no. 20	no. 95	Precision *) of single values in %
Wt%	x	s	s%	x	s	s%			
Na_2O	0.798	0.054	6.9	0.687	0.166	23.3	0.305	0.844	2.1
K_2O	3.790	0.322	8.5	3.414	0.313	9.2	4.155	3.451	2.5
MnO	0.106	0.017	15.8	0.111	0.017	14.9	0.068	0.103	4.4
Fe_2O_3	7.159	0.241	3.37	7.093	0.422	5.9	5.82	5.59	3.3
ppm									
Sc	18.63	0.62	8.7	18.46	0.99	5.3	20.7	15.8	4.7
Cr	170.2	10.6	6.2	170.9	17.3	10.1	131	139	5.9
Ga	27.89	1.94	6.9	27.15	2.53	9.3	29.8	28.8	4.5
Rb	144.5	27.3	18.9	119.6	30.9	25.9	185	98	4.4
Cs	6.72	1.74	25.9	5.63	2.29	40.7	13.9	4	3.6
Ba	609.1	69.2	11.3	663.5	115.7	17.4	373	443	3.7
La	37.2	0.93	2.5	35.5	3.2	9.0	52	21.9	2.1
Nd	0.911	0.106	11.7	0.888	0.141	15.8	1.13	0.534	7.1
Eu	1.312	0.079	6.0	1.269	0.086	6.8	1.82	1.15	4.0
Tb	1.175	0.143	8.2	1.164	0.131	11.2	1.48	1.083	4.0
Yb	2.773	0.304	10.9	2.588	0.387	14.9	3.155	1.239	4.2
Lu	0.349	0.016	4.6	0.343	0.040	11.7	0.371	0.194	5.1
Ta	1.401	0.233	16.6	1.310	0.236	18.0	1.755	1.848	8.2

* Mean standard deviation calculated from 2 duplicate and 2 replicate sample analysis.

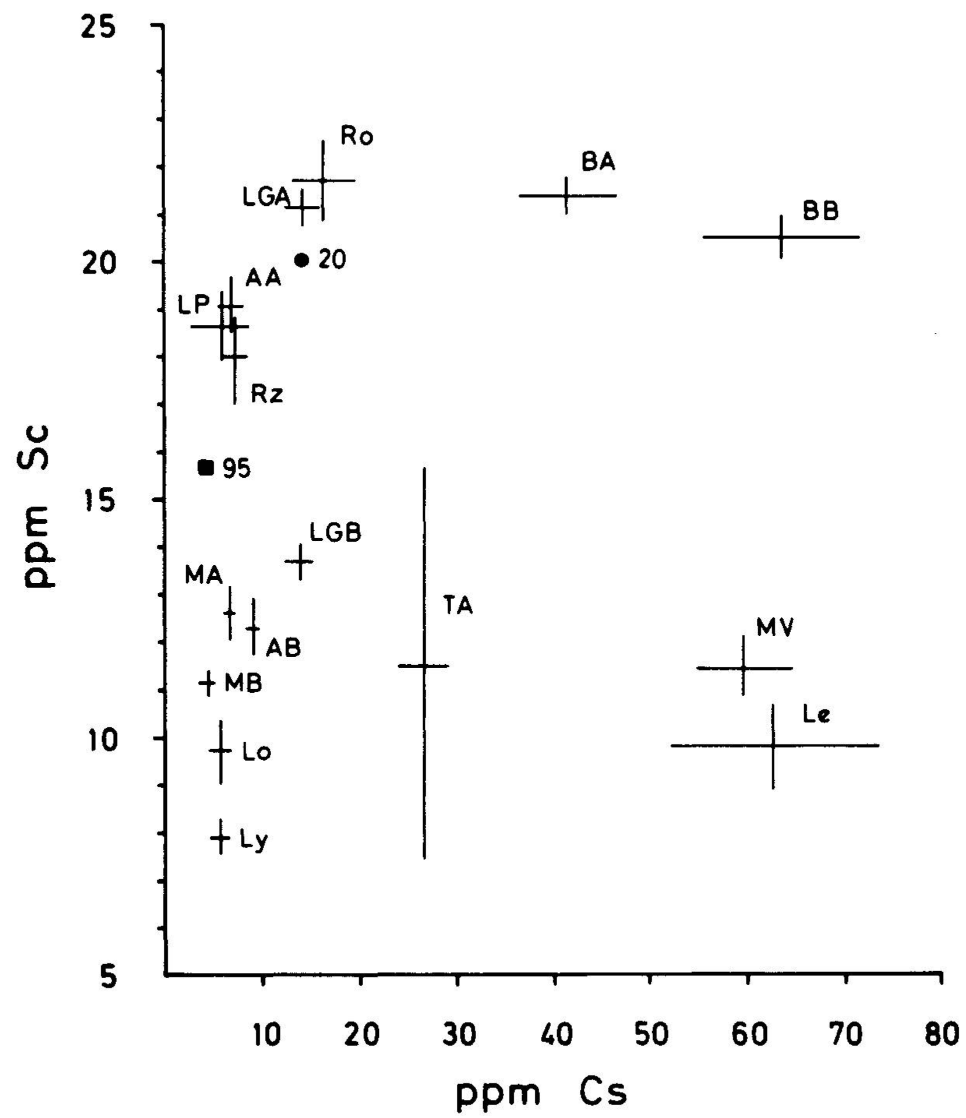

Fig. 2 Sc/Cs correlation diagram for the analysed european Terra Sigillata production centres (NAA). For signs see text. The diagram shows the mean values and the standard deviations.

sherd had no abnormal behaviour during the clustering with the XRF results (Kuepfer and Maggetti 1978).

3.2. EUROPEAN TRACE ELEMENT PATTERN

The Terra Sigillata from La Péniche can well be distinguished by its trace element pattern if some highly discriminative trace elements are plotted into correlation diagrams (e.g. Joron et al., 1977). The published NAA data of the known european workshops summarized in table 2 are taken from the following publications:

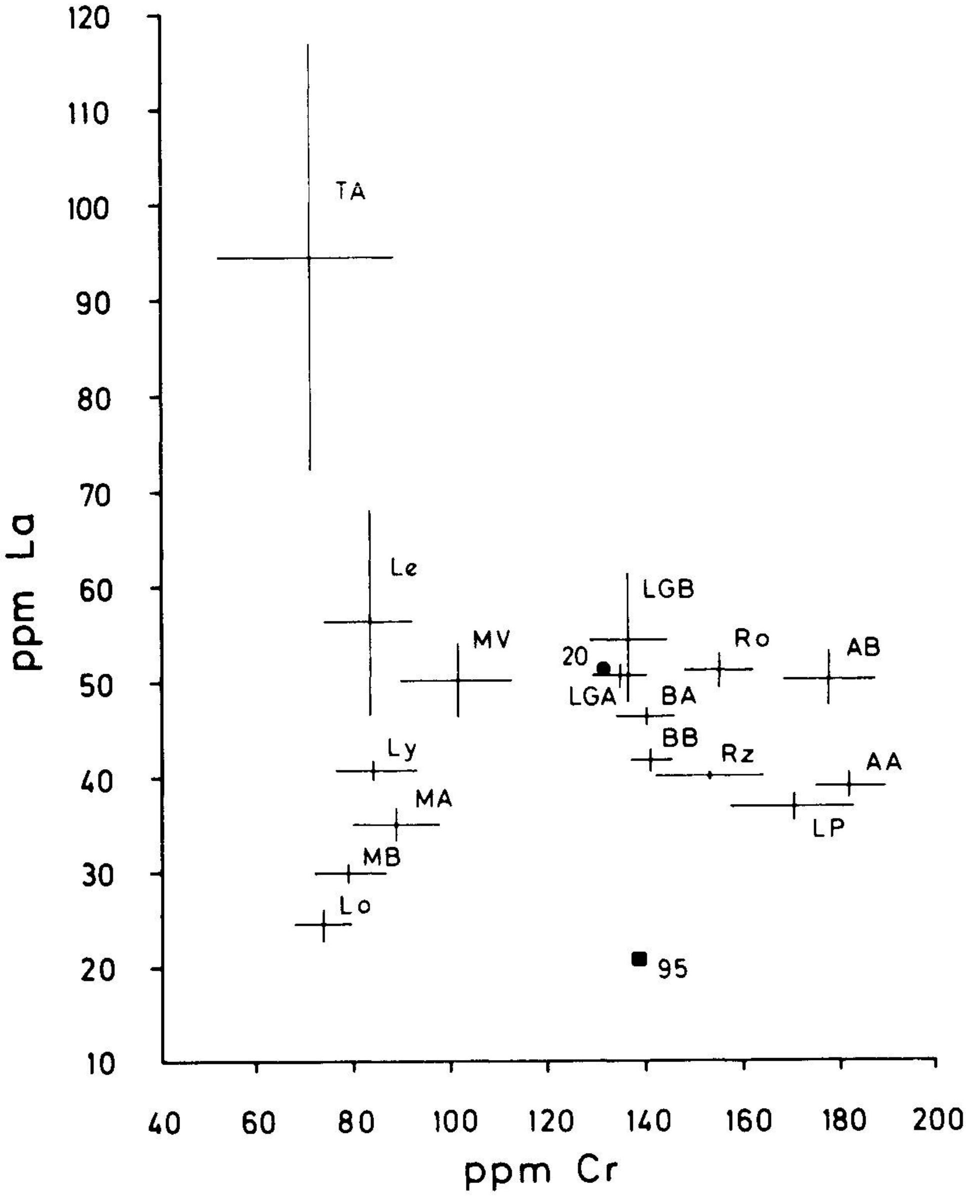

Fig. 3 La/Cr correlation diagram as for fig. 2.

AA	Arezzo	WIDEMANN et al., 1975
AB	Arezzo	JORON 1974
BA	Banassac A	ASARO et al., 1975
BB	Banassac B	ASARO et al., 1975
LGA	La Graufesenque	ASARO et al., 1975
LGB	La Graufesenque	JORON 1974
MA	La Muette A	WIDEMANN et al., 1975
MB	La Muette B	WIDEMANN et al., 1975
LP	La Péniche	this work (48 samples, without 20 + 95)
Ro	Le Rozier	ASARO et al., 1975

Le	Lezoux	JORON 1974
Ly	Lyon	JORON 1974
MV	Martres de Veyre	JORON 1974
Rz	Rheinzabern	SCHNEIDER 1978
TA	Toulon/Allier	JORON 1974
Lo	Loyasse	WIDEMANN et al., 1975

As examples, the Sc/Cs and La/Cr-correlation diagrams are shown (fig. 2, 3). The reference group of La Péniche is close to those of Arezzo and Rheinzabern, but differs markedly from the other groups. A further discrimination against the products of Rheinzabern can be made by comparing K_2O, Lu and Eu, while the differentiation from Arezzo is possible by comparing some major elements as MnO or K_2O.

3.3 COMPARISON OF XRFA AND NAA RESULTS

The comparison of 7 elements (Na_2O, K_2O, Fe_2O_3, MnO, Cr, Rb, Ba) which were determined by both methods yields interesting results (table 3). If the analytical results of both methods are compared, the NAA gives in general higher values for Ba, K_2O, MnO, Cr, Rb, Fe_2O_3 and lower for Na_2O. This is also true for the mean values, where the differences can differ up to 15%. The standard deviations are significantly higher for the NAA for Fe_2O_3 and Ba. These differences are related with the method and must be taken in mind if results of different laboratories and/or different methods are compared (for general discussion of these problems see SCHNEIDER 1978).

Table 2. European trace element pattern (ppm, NAA) for the analysed Terra Sigillata workshops.

	Arezzo A				Arezzo B				Banassac A				Banassac B			
	x	s	s%	n	x	s	s%	n	x	s	s%	n	x	s	s%	n
Sc	19.06	0.59	3.1	23	12.32	0.56	4.5	11	21.41	0.34	1.6	10	20.49	0.48	2.3	12
Cr	182	6	0.3		178.45	8.68	4.9	11	140.	6	4.3		141	4	2.8	
Co	22.55	0.86	3.8		30.36	19.03	62.7	11	19.94	1.04	5.2		19.89	0.58	2.9	
Ni	98	15	15.3		79.33	3.49	4.4	6	n.d.				n.d.			
Cu	n.d.				77.17	52.53	68.1	6	n.d.				n.d.			
Zn	139	7	5.0		181.5	50.48	27.8	6	n.d.				n.d.			
As	6.5	1	15.4		n.d.				n.d.				n.d.			
Rb	132	16	12.1		179.17	22.23	12.4	6	n.d.				n.d.			
Sb	0.67	0.07	10.4		n.d.				n.d.				n.d.			
Cs	6.98	1.1	15.7		7.79	1.43	18.3	11	40.9	5.2	12.7		62.9	9.25	14.7	
Ba	435	36	8.3		n.d.				n.d.				n.d.			
La	39.4	1.1	2.8		50.25	3.5	6.9	4	46.5	1.1	2.4		42.6	1.5	3.5	
Ce	80.8	3	3.7		84.17	4.31	5.1	6	90.7	1.6	1.8		83.8	4.1	4.9	
Sm	6.56	0.20	3.1		6.6	0.32	4.8	4	6.86	0.11	1.6		6.27	0.21	3.4	
Eu	1.46	0.05	3.7		1.63	0.2	12.1	6	1.53	0.06	3.7		1.49	0.1	6.8	
Dy	5.39	0.21	3.9		n.d.				5.47	0.22	4		4.91	0.23	4.7	
Yb	2.79	0.08	2.9		2.9	0.18	6.3	4	2.95	0.01	0.3		2.71	0.06	2.2	
Lu	0.4	0.02	4.2		0.85	0.05	6.1	5	0.43	0.02	4.6		0.4	0.02	5	
Hf	3.95	0.12	3		n.d.				4.59	0.14	1.8		4.34	0.22	5.1	
Ta	1.24	0.04	3.1		n.d.				1.17	0.02	1.4		1.08	0.05	4.7	
Th	13.44	0.47	3.5		12.09	1.63	13.4	11	13.53	0.44	3.2		13.04	0.6	4.6	
U	2.7	0.09	3.3		n.d.				3.01	0.11	3.6		2.92	0.05	1.7	

n.d. = not determined

Table 2 (continued)

	La Graufesenque A				La Graufesenque B				La Muette A				La Muette B			
	x	s	s%	n	x	s	s%	n	x	s	s%	n	x	s	s%	n
Sc	21.22	0.41	1.9	34	13.73	0.41	2.9	11	12.6	0.65	5.1	14	11.07	0.33	2.9	4
Cr	135	6	4.1		137.3	7.85	5.7	11	89	9	10.1		79	7	8.9	
Co	17.31	1.33	7.5		17.9	4.26	23.8	11	14.3	0.78	5.4		12.87	0.19	1.5	
Ni	n.d.				56.4	4.83	20.3	5	58	15	25.9		58	16	27.6	
Cu	n.d.				33.8				n.d.				n.d.			
Zn	n.d.				197.6	99.35	50.3	5	83	8	9.6		85	7	8.2	
As	n.d.				n.d.				15.1	2	13.2		27	3.9	14.4	
Rb	n.d.				n.d.				125	9	7.2		82	21	25.6	
Sb	n.d.				n.d.				1.25	0.12	9.6		1.42	0.1	7.0	
Cs	14.33	0.48	3.3		13.05	1.74	13.3	11	6.5	0.47	7.3		3.68	0.61	16.6	
Ba	n.d.				n.d.				415	68	16.4		470	26	5.5	
La	50.5	1.5	3		54.6	2.61	4.8	5	34.7	1.7	4.9		29.7	0.5	1.7	
Ce	98.5	3	3		85	7.11	8.4	5	68.4	3.8	5.5		56.8	0.9	1.6	
Sm	7.58	0.23	3		7.14	0.09	1.2	5	6.1	0.28	4.6		6.56	0.2	3.1	
Eu	1.66	0.62	3.7		n.d.				1.38	0.07	5.2		1.12	0.02	1.6	
Dy	5.92	0.25	4.2		n.d.				5.3	0.28	5.3		4.58	0.31	6.8	
Yb	3.15	0.12	3.8		2.98	0.3	10.2	5	2.7	0.14	5.2		2.25	0.05	2.2	
Lu	0.45	0.02	4.4		n.d.				0.39	0.02	6.4		0.31	0.02	6.9	
Hf	5.06	0.3	5.9		n.d.				4.11	0.22	5.3		3.06	0.13	4.2	
Ta	1.27	0.04	2.9		n.d.				0.98	0.04	4.2		0.77	0.02	2.3	
Th	n.d.				13.59	1.09	8.03	11	12.1	0.54	4.5		10.48	0.13	1.2	
U	3.09	0.1	3.2		n.d.				2.6	0.13	5		2.61	0.22	8.4	

Table 2 (continued)

	Le Rozier				Lezoux				Lyon				Martres de Veyre			
	x	s	s%	n	x	s	s%	n	x	s	s%	n	x	s	s%	n
Sc	21.7	0.97	4.5	10	9.85	0.97	9.8	22	7.9	0.47	5.9	11	11.43	0.7	6.1	21
Cr	155	7	4.5		83.45	10.01	12	22	84.55	8.95	10.6	11	103.1	10.63	10.3	
Co	17.81	1.7	9.5		15.57	5.7	36.6	22	14.05	1.08	7.7	11	9.52	0.7	7.4	
Ni	n.d.				36.8	4.54	12.3	22	47.2	5.12	10.8	5	n.d.			
Cu	n.d.				21.55	6.53	30.2	22	19.8	4.21	21.2	5	n.d.			
Zn	n.d.				155.1	23.14	14.9	21	85	43.97	51.7	5	n.d.			
As	n.d.				n.d.				n.d.				n.d.			
Rb	n.d.				348.48	40.49	11.6	21	144.2	15.59	10.8	5	333.19	29.92	8.9	
Sb	n.d.				n.d.				n.d.				n.d.			
Cs	16.04	3.25	20.2		62.27	10.44	16.8	22	6.27	0.47	7.4	11	59	5.1	8.7	
Ba	n.d.				n.d.				n.d.				n.d.			
La	51.18	1.91	3.7		56.52	11.47	20.3	21	40.67	1.53	3.8	3	50.14	3.87	7.7	
Ce	5.33	0.2	3.7		84.27	20.41	24.2	22	69.8	4.09	5.8	5	75.86	7.22	9.5	
Sm	7.43	0.18	2.4		6.01	0.87	14.4	21	6.17	0.12	1.9	3	5.51	0.83	15.1	
Eu	1.74	0.07	4		1.41	0.19	13.2	22	1.5	0.07	4.7	5	1.38	0.12	9.1	
Dy	5.92	0.2	3.4		n.d.				n.d.				n.d.			
Yb	3.08	0.06	1.9		2.65	0.64	24.1	21	3.07	0.29	9.4	3	2.59	0.36	13.9	
Lu	n.d.				0.37	0.08	21.7	21	0.77	0.07	9.5	5	0.37	0.06	16.6	
Hf	5.33	0.2	3.7		n.d.				n.d.				n.d.			
Ta	1.38	0.09	6.6		n.d.				n.d.				n.d.			
Th	15.2	0.71	4.7		20.84	3.76	18	22	10.89	1.6	14.6	11	16.31	1.33	8.2	
U	2.92	0.09	3.1		n.d.				n.d.				n.d.			

*) Mineralogisch-Petrographisches Institut der Universität, Wilhelmstrasse 56, D-7400 Tübingen 1.

Table 2 (continued)

	Rheinzabern				Toulon/Allier				Loyasse				La Péniche			
	x	s	s%	n	x	s	s%	n	x	s	s%	n	x	s	s%	n
Sc	18	1.8	10	11	11.47	4.18	36.4	20	9.8	0.69	7	5	18.6	0.72	3.9	48
Cr	153	10.7	7	8	72.95	17.51	24		73	6	8		171	13.7	8	
Co	14	2.1	15	11	11.19	4.11	36.7		10.27	0.82	7.9		n.d.			
Ni	n.d.				n.d.				40	11	27		n.d.			
Cu	n.d.				n.d.				n.d.				n.d.			
Zn	n.d.				n.d.				73	8	11		n.d.			
As	n.d.				n.d.				25.9	10.2	39.4		n.d.			
Rb	147	52.9	36	4	270.57	23.32	8.6		92	19	21		132	31.6	23.9	
Sb	n.d.				n.d.				1.46	0.4	27.4		n.d.			
Cs	7	1.05	15	11	26.33	2.67	10.1		5.3	0.95	22.1		6.19	2.1	33.9	
Ba	n.d.				n.d.				731	198	27		642.1	96.4	15	
La	40			2	94.3	22.99	24.4		27.4	1.7	6.2		36.64	1.45	3.9	
Ce	70	7	10	9	129.1	31.5	24.4		54	3.6	6.7		n.d.			
Sm	7.3			2	9.01	2.91	32.3		4.69	0.28	5.9		n.d.			
Eu	1.6	0.18	11	11	1.83	0.49	26.9		1.017	0.059	5.8		1.292	0.084	6.5	
Dy	n.d.				n.d.				4.04	0.44	10.9		n.d.			
Yb	n.d.				4.96	1.32	26.5		2.19	0.11	5		2.705	0.295	10.9	
Lu	0.6	0.12	20	11	0.58	0.13	21.9		0.328	0.021	0.1		0.349	0.022	6.3	
Hf	3.6	0.54	15	9	n.d.				4.12	0.47	11.4		n.d.			
Ta	n.d.				n.d.				0.785	0.048	6.1		1.342	0.227	16.9	
Th	16	0.64	4	8	43.95	13.73	31.2		9.7	0.62	6.4		n.d.			
U	7.3			2	n.d.				2.33	0.33	14.2		n.d.			
Ga	n.d.				n.d.				n.d.				27.47	2.3	8.4	
Nd	n.d.				n.d.				n.d.				0.907	0.114	12.5	
Tb	n.d.				n.d.				n.d.				1.171	0.136	11.6	

Table 3. Comparison of the chemical analysis by XRF and NA for the Terra Sigillata from La Péniche. XRF (TS, n = 41, TH, n = 47), NAA (TS, n = 23, TH, n = 25).

		XRFA		NAA	
Wc%		x	s	x	s
Na_2O	TS	0.91	0.057	0.799	0.054
	TH	0.79	0.17	0.7	0.16
K_2O	TS	3.41	0.25	3.79	0.32
	TH	2.8	0.46	3.16	0.45
Fe_2O_3 *	TS	6.3	0.12	7.15	0.24
	TH	6.42	0.32	7.08	0.44
MnO	TS	0.087	0.003	0.103	0.008
	TH	0.091	0.006	0.107	572
96		663	115		

* Fe tot as Fe203

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