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Application of ICP-MS trace element analysis in study of ancient Chinese ceramics

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Abstract **Thirty-nine trace elements of the Song-Yuan period (960—1368 AD) porcelain bodies from Cizhou, Jizhou and Longquanwu kilns were analyzed with ICP-MS, a technique rarely used in Chinese archaeometry, to investigate its potential application in such studies. Trace element compositions clearly reflect the distinctive raw materials and their mineralogy at the three kilns and allow their products to be distinguished. Significant chemical variations are also observed between Yuan and Song-Jing dynasties samples from Cizhou as well as fine and coarse porcelain bodies from Longquanwu. In Cizhou, porcelains of better quality which imitate the famous Ding kiln have trace element features distinctive from ordinary Cizhou products, that indicates geochemically distinctive raw materials were used and which possibly also underwent extra refining prior to use. The distinct trace element features of different kilns and the various types of porcelains from an individual kiln can be interpreted from a geochemical perspective. ICP-MS can provide a large amount of valuable information about ancient Chinese ceramics as it is capable of analyzing >40 elements with a typical of precision < 2%.**

Keywords: Cizhou kiln, Jizhou kiln, Longquanwu kiln, Ding kiln, ancient ceramics, Chinese Song-Yuan period, trace elements, ICP-MS.

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Chemical analysis plays a significant role in the study of ancient ceramics[1,2]. Most ancient Chinese kilns used clays mined from the local areas and differences in the geochemistry and mineralogy of these raw materials may be expected. The chemical composition of ceramics may also be influenced by production techniques, such as processing by washing and mixing of different sorts of raw materials. These may also vary from kiln to kiln and even change over time. Thus the raw materials and production techniques used by a kiln may give its finished products a characteristic chemical signature allowing their provenance and possibly even their age to be determined[3,4].

Among the analytical methods commonly used for ancient Chinese ceramics are neutron activation analysis (NAA)[5], X-ray fluorescence (XRF)[6], and proton-induced X-ray emission (PIXE)[7]. Compared with other techniques, inductively coupled plasma mass spectrometry (ICP-MS) is relatively new and particularly advantageous in trace element analysis. It is capable of simultaneously and rapidly analysing over 40 elements with high precision and accuracy, and with excellent detection limits. Only a small amount of sample is required (10—100 mg) for solution analysis, and sample preparation is relatively simple[8]. It is being applied more commonly in western countries for the study of ancient ceramics[9], yet has only very rarely been used in Chinese archaeometric research[10].

As the trace element compositions of raw materials are likely to be more variable than their major elements, the large number of trace elements that can be determined by ICP-MS increases the likelihood of finding distinctive chemical signatures in kiln products. Here we demonstrate this advantage in a study of ceramics produced at three ancient Chinese kilns.

1 Samples

We have analysed the bodies of 58 porcelain shards from Jizhou, Cizhou and Longquanwu kilns, which were very important producers of white and black porcelains during the Chinese Song-Yuan period (960—1368 AD). 11 shards are from Jizhou kiln (located in Ji' an City, Jiangxi Province) and have external black glaze[11]. 24 white glazed shards are from Lonquanwu kiln (located in Mentougou, Beijing, Hebei Province) and these can be classified as either coarse or fine according to the grain size, porosity and number of visible quartz grains of the porcelain bodies[12,13]. 23 Cizhou shards are from the Guantai production centre in Cixian County, Hebei Province. These have either white or black external glazing. Cizhou porcelains from Guantai are divided into four periods based on archaeological-stratigraphic and morphological information[14]. The four periods are approximately equivalent with the early and mid Northern Song Dynasty, late Northern Song to early Jin Dynasty, mid to late Jin Dynasty, and Yuan Dynasty, respectively. 5 shards are from periods 1 and 4, 10 from period 2 and 3 from period 3.

During the Song-Yuan period porcelains from different kilns often look similar due to a tendency to imitate products from other kilns, thus making it difficult to identify their place of origin. For example, the bodies of most Cizhou porcelains are thick, coarse and dark in colour, and are ordinary goods for a popular market. However, among Cizhou products is a specific type of porcelain of exquisite workmanship whose bodies are overall thinner and finer and, for those covered with a white glaze, also much whiter than the bodies of the ordinary Cizhou products. These are imitations of the products of the contemporary Ding kiln in Quyang County, Hebei Province which are famous for their exquisite workmanship and thin, fine and white porcelain bodies[15]. Among our analysed Cizhou samples are 5 white glazed imitation Ding porcelains, which belong to Guantai period 2. The Ding kiln also heavily influenced the Longquanwu kiln while the influences of both the Cizhou and Ding kilns reached as far as the Jizhou kiln[16,17].

2 Analytical methods

The glaze of the porcelain shards was completely removed by grinding on a diamond lap. The porcelain bodies were cleaned with 18.2 megaohm water in an ultrasonic bath and then dried. Approximately 50—100 mg of each sample were weighed and digested with distilled HF and HNO_3 acids using high pressure teflon bombs in an oven at 180℃ for 60 h to ensure dissolution of all minerals, particularly refractory minerals such as zircon. The samples were then dried down and redissolved in distilled HCl in the oven at 180℃ overnight. The solutions are transferred to a polystyrene tube and centrifuged. Any remaining solids are transferred back to the bombs and dissolved in HCl overnight in the oven. If dissolution is complete the solutions are combined and dried down and the sample converted to nitrates by twice adding HNO_3 and drying down. Other sample preparation and analytical procedures used are identical to those of Eggins et al.[8], except that Tm was not used as an internal standard and W-2, an international rock standard, was used as the calibration standard. Our preferred concentrations for W-2 are shown in Table 1. The accuracy and precision of our technique can be assessed from the measured concentrations and relative standard deviations for rock standards BHVO-1 (an average of 203 analyses of 54 digestions analysed over five years) and G-2 (an average of 9 analyses of 3 digestions) which are shown in Table 1. The samples were analysed with a Fisons PQ2+ ICP-MS at the Advanced Centre for Queensland University Isotope Research Excellence (ACQUIRE).

3 Results and discussion

ICP-MS results of 39 trace elements for representative samples are shown in Table 1 and the variations of selected elements for all 58 analysed samples are illustrated in Fig. 1.

Table 1 and Fig. 1 demonstrate that porcelain bodies from Cizhou, Longquanwu and Jizhou have distinctive concentrations of numerous trace elements (eg. Sr, Ba, Rb, W, Ta, Nb, Be, U, Li) and element ratios (Nb/Ta, Y/Ho), which allow products from one kiln to be distinguished from one or more of the other kilns. These differences reflect the distinctive mineralogy and geochemistry of the raw materials used at the three kilns. Longquanwu porcelains are distinct from the other kilns due to their very high and very variable Sr concentrations and low Rb/Sr ratios of less than 0.5. They also have high Ba contents and some samples have Eu/Eu* > 1 indicating that they

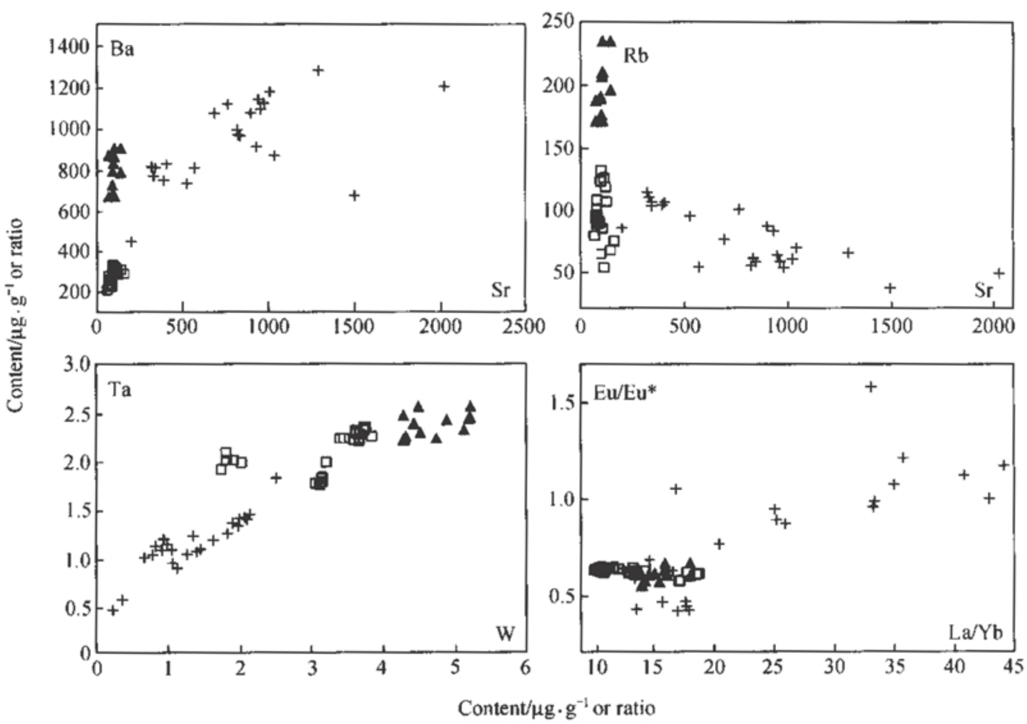

Fig. 1. Comparison of trace elements of all porcelain body samples from Cizhou, Jizhou and Longquanwu. Square: Cizhou, triangle: Jizhou, cross: Longquanwu. Eu/Eu* is a measure of Eu anomalies on chondrite-normalized REE diagrams. Values greater than 1 indicate a positive anomaly while values less than 1 indicate a negative anomaly.

have positive Eu anomalies on chondrite-normalized rare earth element (REE) diagrams (Fig. 1). Very high Sr concentrations and positive Eu anomalies are not typical of naturally formed clay minerals, and suggest that feldspar was added to the clay during production, a suggestion also made by Chen et al. based on petrographic observations and the high Na content of Longquanwu porcelains[18]. During crystallization feldspar preferentially incorporates Eu into its structure relative to neighbouring REE and it can also have high Sr and Ba contents. The low Sr, Ba and Rb contents of Cizhou samples can be related to the use of the clay abundant in the Cizhou area, which is dominated by kaolinite[19], a mineral expected to be low in Rb, Sr and Ba contents. In contrast, Jizhou porcelain stone (*cishi*) or weathered porcelain stone was used for the porcelain bodies[20—22], which is dominated by sericite[23], a type of mica typically rich in Rb and Ba but low in Sr contents, which causes the high Rb and Ba and low Sr contents of Jizhou porcelains.

Apart from the distinctive trace element signatures of the three kilns, differences in trace element features are also observed within porcelain groups from one individual kiln. Longquanwu samples with coarse bodies (lqw-c) tend to have higher Rb, W, Y and REE contents and lower Sr and Li contents and La/Yb and Eu/Eu* ratios than those with fine bodies (lqw-f, Table 1). This is consistent with previous observations that fine and coarse bodied Longquanwu porcelains have different major and minor element features such as in Al and Fe contents[13].

Previous EDXRF and WDXRF major and trace element analysis of Cizhou porcelains have shown that imitation Ding porcelain bodies have lower Fe and Ti contents than ordinary Cizhou products. It was proposed that higher degrees of refining of the raw material were carried out when producing imitation Ding bodies than for ordinary products and that this would cause a decrease in the content of ferrous mineral particles and Fe contents and hence improve the whiteness of the bodies[24,25]. More refining may also remove more coarse fractions of mineral particles such as quartz and other impurities and achieve a porcelain body finer grained than ordinary products. Our ICP-MS analyses confirm that imitation Ding porcelain bodies (gt-b1 – gt-b3, Table 1) do have lower Ti, but that they also have markedly lower contents of Sc, V, Ni, Cr, U and W and higher La/Yb and Nb/Ta than ordinary Cizhou products (Fig. 2). Sometimes the contrast between these Cizhou sub-groups even exceeds that between Jizhou or Longquanwu porcelains and ordinary Cizhou products.

Heavy Fe and/or Ti rich minerals such as magnetite, ilmenite and rutile are likely to preferentially host transition elements such as Sc, V, Ni and Cr as well as Nb and Ta, but exclude REE. Thus, removal of these mineral phases due to extra refining would also cause a decrease in Nb, Ta and transition element contents as well as in Fe and Ti contents, while La/Yb ratios would not be affected. In addition, the Nb/Ta ratio of the more refined product would be expected to also remain unchanged or perhaps decrease. As imitation Ding bodies actually have much higher Nb/Ta and La/Yb ratios and either the same or higher Nb contents compared to ordinary Cizhou bodies (Table 1), greater refining of the raw material used to make ordinary Cizhou products by removal of Fe and/or Ti rich minerals will not produce the compositions of imitation Ding bodies. Therefore we suggest that the raw materials used for imitation Ding porcelain bodies are geochemically distinct from those used for ordinary Cizhou products. Presumably a different source of raw materials more suitable for producing the high quality imitation Ding products was selected. This would enable fine quality porcelains to be produced without drastic increases in production costs that excessive refining would cause.

Our ICP-MS analysis also detects subtle differences between ordinary Cizhou products from period 4 (gt-d) and periods 1—3 at Guantai (Fig. 2). Excluding the imitation Ding porcelains, ordinary Cizhou products from period 4 (Yuan Dynasty) tend to have lower contents of Ti, Cr, Zr, Hf, Th, Nb, and Ta and are higher in Rb, Ba and total REE contents than those from periods 1—3 (gt-a to gt-c, Table 1). The ordinary products from the first three periods (Northern Song and Jin Dynasties) are not readily distinguished. Such trace element characteristics are a reflection of the temporal change in the development history of porcelain production at Guantai. Whilst there are not significant quality changes in bodies of ordinary Guantai porcelains from the first three periods, period 4 saw the obvious decline of porcelain production at Guantai. With an evident decrease in overall product quality, the porcelain bodies became noticeably coarser. In view of this observation, ICP-MS trace element compositions may potentially assist in dating ceramics. Apart from the white imitation Ding porcelains, no noticeable trace element differences are observed between the ordinary Cizhou products with a white or black glaze. This indicates that there was not much difference between the raw materials for ordinary Cizhou porcelains of white or black glaze, as also observed in previous studies[26].

4 Conclusions

The 39 trace elements from ICP-MS analyses of porcelain bodies from Cizhou, Jizhou and Longquanwu provide a large amount of valuable information concerning the provenance, raw materials and their sources, production technology and dating of these ceramics. With other analytical methods, many of the elements and ratios that distinguish different Cizhou products are difficult or impossible to analyse (eg. W, Nb/Ta, U). Capable of analysing a large number of elements with high precision and accuracy, ICP-MS is a promising method for ancient

Table 1 ICP-MS data (in $\mu g \cdot g^{-1}$) of standards and some porcelain body samples from Cizhou, Longquanwu and Jizhou kilns

	W-2	BHVO-1	%RSD	G-2	%RSD	gt-a1	gt-a2	gt-a3	gt-b1	gt-b2	gt-b3	gt-b9	gt-b10	gt-b11	gt-c1	gt-c2	gt-c3	gt-d1	gt-d2	gt-d3	lqw-c3	lqw-c5	lqw-f15	lqw-f24	jz2	jz6
Li	9.16	4.70	1.3%	33.48	1.1%	102.2	144.0	107.5	96.7	91.9	98.8	123.8	137.5	137.9	145.9	144.8	133.8	118.4	102.0	103.8	23.4	11.41	81.4	315	25.9	14.67
Be	0.617	0.908	2.1%	2.46	3.9%	2.15	2.11	2.01	3.45	3.08	3.36	2.34	2.02	2.28	2.64	2.60	2.71	2.73	2.54	3.03	2.32	2.27	3.20	3.51	10.29	6.72
Sc	36.1	31.8	1.6%	5.0	41.6%	24.2	23.3	21.0	19.2	19.4	20.0	24.0	23.3	25.6	26.9	24.7	23.9	26.4	25.7	26.0	19.4	21.5	13.5	17.4	17.08	20.7
Ti	6355	16395	3.1%	2932	1.2%	7923	8064	7394	6002	6068	6174	7865	7910	8097	8706	8282	6380	6765	6846	6607	6460	6438	7021	7523	5987	6010
V	262	310	1.3%	34	0.7%	201	217	181.9	102.0	100.9	101.3	224	202	226	222	211	177.4	194.4	191.4	186.1	148.6	138.2	175.9	174.4	101.3	112.4
Cr	93	295	2.1%	7.0	1.9%	154.1	151.1	127.8	65.7	68.9	70.6	146.8	145.6	167.2	164.6	151.4	132.3	114.1	112.6	110.7	83.1	74.2	117.8	100.0	84.7	95.4
Co	44.5	45.0	1.4%	4.6	5.1%	4.30	4.62	3.07	4.22	3.59	5.04	5.31	4.07	4.68	7.36	5.13	4.43	4.75	5.41	11.22	3.82	2.16	2.49	6.0	3.23	2.04
Ni	70	116	2.3%	3	18.6%	25.2	23.2	18.4	13.5	13.6	15.8	28.3	22.6	27.0	28.1	24.5	30.0	20.6	19.4	28.7	12.60	5.87	6.60	13.0	16.32	12.95
Cu	103	142	5.6%	10	3.9%	11.5	13.6	6.2	12.9	11.2	12.9	8.8	8.4	7.2	14.9	13.4	19.6	21.1	17.8	19.4	19.9	17.9	12.4	7.8	15.9	18.2
Zn	77	106	5.4%	92	9.5%	8.87	5.82	16.1	26.3	21.2	37.6	9.78	6.79	4.36	19.62	21.4	20.3	25.9	20.5	41.2	27.0	12.2	8.2	8.3	21.6	14.7
Ga	17.4	21.1	1.1%	22.8	1.0%	40.9	39.8	39.9	41.6	41.0	37.8	38.6	40.0	41.6	40.8	42.1	39.1	34.9	33.8	32.6	30.7	30.8	39.7	41.1	28.8	33.4
Rb	19.80	9.31	0.8%	169.3	0.7%	91.4	95.7	80.6	68.5	76.1	86.2	97.0	90.1	94.5	91.1	92.8	107.9	126.6	124.5	124.3	110.2	108.0	76.6	55.0	176.6	207.1
Sr	194.8	396	0.5%	474	0.7%	87.4	74.5	64.1	141.7	159.8	103.9	73.4	73.6	71.2	80.3	74.5	123.6	109.3	95.8	96.0	329	346	689	823	102.1	103.5
Y	20.1	24.6	0.7%	9.32	1.2%	41.8	42.4	37.9	39.6	37.3	43.2	41.9	41.1	44.3	42.7	44.8	51.9	53.9	46.1	47.3	47.0	43.9	19.9	14.0	47.9	41.3
Zr	87.9	170.0	1.4%	330	1.9%	411	418	384	382	366	374	417	411	412	432	407	392	321	327	313	356	347	339	365	348	322
Nb	7.28	18.47	1.0%	12.40	1.2%	30.6	30.4	29.4	30.5	30.9	30.1	29.0	30.7	29.4	31.0	29.8	25.1	23.7	24.1	23.1	20.4	20.1	18.8	19.4	25.6	26.2
Sn	1.95	2.22	12.7%	1.97	2.8%	6.55	8.05	7.76	2.25	1.62	1.82	6.85	6.57	6.85	3.23	6.10	6.95	6.13	2.37	2.16	4.35	4.08	3.33	2.94	11.72	13.14
Cs	0.888	0.097	2.2%	1.358	1.2%	8.62	9.39	8.14	6.37	6.53	6.30	9.24	8.53	9.61	8.49	10.02	10.96	9.87	9.59	9.01	6.29	6.32	8.04	6.24	13.18	11.92
Ba	169.7	131.8	0.7%	1887	0.9%	231	231	210	311	290	322	235	232	233	254	230	286	321	325	329	823	813	1075	995	688	837
La	10.52	15.43	0.7%	88.6	0.7%	57.9	53.6	46.8	81.3	75.9	82.9	51.3	50.0	62.6	50.0	55.5	76.6	77.4	70.8	69.6	65.3	75.7	62.2	63.8	78.3	67.4
Ce	23.2	38.2	0.7%	164.3	0.7%	115.3	110.2	95.4	168.8	158.1	175.1	103.7	100.9	122.3	101.7	113.0	160.1	162.9	148.9	147.6	137.0	162.8	117.4	111.8	161.3	130.7
Pr	3.03	5.46	0.7%	16.83	0.5%	12.3	11.8	10.0	18.2	17.2	18.9	11.5	10.6	12.7	11.0	12.4	17.5	17.5	16.4	16.8	15.9	18.7	12.6	13.0	17.9	16.2
Nd	12.91	24.7	0.6%	53.1	0.7%	41.7	41.2	33.8	62.3	58.8	65.1	41.5	36.4	42.7	38.4	43.6	60.4	58.6	57.2	59.3	58.4	66.3	43.7	43.9	63.5	59.7
Sm	3.27	6.13	0.9%	7.14	1.2%	7.34	7.43	6.34	10.73	10.04	11.52	7.82	6.52	7.43	7.11	8.02	11.05	10.83	10.87	11.52	11.29	10.78	7.52	7.06	11.99	12.01
Eu	1.094	2.073	0.9%	1.336	1.6%	1.437	1.452	1.258	1.884	1.754	1.926	1.509	1.269	1.449	1.407	1.568	2.090	2.036	2.019	2.117	1.521	1.419	1.778	1.703	2.178	2.371
Gd	3.710	6.292	0.9%	4.000	1.8%	6.26	6.47	5.49	8.15	7.62	9.05	6.95	5.81	6.39	6.42	7.28	9.40	9.09	8.82	9.47	10.14	8.91	5.19	4.19	9.89	9.71
Tb	0.65	0.95	0.8%	0.48	1.6%	1.107	1.105	0.992	1.264	1.194	1.360	1.167	1.044	1.141	1.119	1.247	1.563	1.571	1.415	1.540	1.58	1.47	0.71	0.552	1.54	1.41
Dy	3.81	5.30	0.9%	2.19	1.6%	7.19	7.22	6.44	7.44	6.93	8.12	7.29	6.90	7.60	7.21	7.77	9.49	9.85	8.62	9.25	9.16	8.70	4.00	3.04	8.93	7.83
Ho	0.803	0.101	0.8%	0.372	1.2%	1.582	1.571	1.391	1.525	1.442	1.688	1.553	1.515	1.659	1.587	1.690	1.983	2.061	1.810	1.912	1.880	1.731	0.820	0.625	1.805	1.563
Er	2.22	2.53	0.9%	0.901	1.1%	4.56	4.54	4.08	4.28	4.06	4.77	4.51	4.41	4.84	4.68	4.97	5.76	5.98	5.14	5.41	5.15	4.65	2.32	1.80	5.00	4.34
Tm	0.327	0.343	1.3%	0.122	0.9%	0.743	0.743	0.658	0.662	0.633	0.748	0.715	0.719	0.782	0.749	0.780	0.896	0.923	0.808	0.839	0.762	0.684	0.363	0.281	0.759	0.657
Yb	2.06	2.012	1.0%	0.731	1.3%	4.97	4.97	4.40	4.33	4.09	4.83	4.71	4.71	5.16	4.92	5.13	5.85	5.95	5.24	5.39	4.80	4.26	2.39	1.92	4.85	4.22
Lu	0.301	0.278	1.3%	0.102	1.7%	0.740	0.741	0.664	0.639	0.603	0.710	0.702	0.705	0.762	0.748	0.777	0.888	0.882	0.774	0.795	0.703	0.606	0.356	0.287	0.712	0.635
Hf	2.36	4.35	1.3%	7.83	2.3%	11.10	11.28	10.31	9.96	9.61	9.99	11.39	11.06	11.08	11.75	11.11	10.70	8.87	9.01	8.63	9.49	9.50	8.48	8.88	9.28	8.70
Ta	0.454	1.155	1.3%	0.800	1.3%	2.32	2.28	2.24	2.02	2.09	1.99	2.22	2.36	2.23	2.32	2.29	1.99	1.82	1.84	1.77	1.46	1.43	1.23	1.21	2.25	2.29
W	0.240	0.193	3.3%	0.096	1.6%	3.62	3.69	3.42	1.82	1.82	2.03	3.61	3.74	3.68	3.75	3.63	3.21	3.15	3.15	3.13	2.14	2.06	1.34	0.94	4.33	4.53
Pb	7.53	2.02	3.8%	30.5	2.2%	24.3	21.8	18.16	39.8	31.8	36.7	23.2	19.9	24.2	23.2	20.7	42.3	41.9	35.2	29.1	16.8	16.3	9.6	11.0	18.2	12.0
Th	2.10	1.198	1.1%	23.8	1.2%	27.6	27.2	25.6	24.4	24.9	24.5	27.6	26.0	27.9	28.9	26.6	26.0	24.2	24.2	23.6	18.6	19.2	17.2	18.1	22.0	23.2
U	0.505	0.427	1.3%	1.886	1.8%	6.44	6.27	6.03	5.04	4.96	4.83	6.84	6.05	7.16	6.53	6.45	8.36	6.99	6.06	5.98	3.76	3.55	3.53	3.51	7.14	6.72

Sample series gt-a to gt-d refer to Cizhou samples from periods 1 to 4 at Guantai site, respectively. Among Period 2 samples gt-b1 to gt-b3 are imitation Ding porcelains. Lqw-c and lqw-f refer to coarse and fine Longquanwu porcelain bodies, respectively. Jz refers to Jizhou samples. W-2, G-2 and BHVO-1 are our laboratory standards. %RSD denotes relative standard deviation. See the text for discussion.

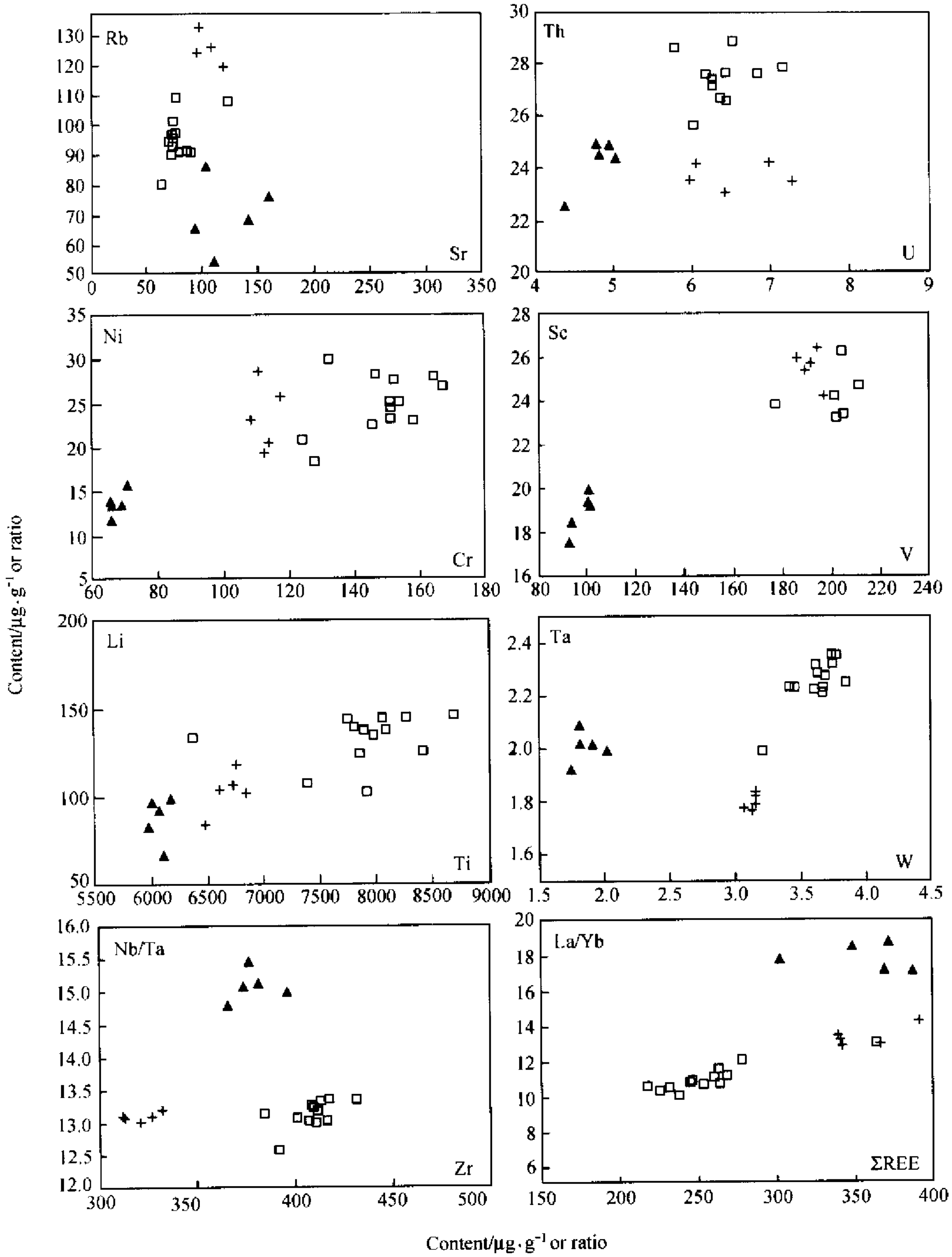

Fig. 2. Trace elements plots of all Cizhou porcelain body samples of different types and different periods from Guantai site. Triangle: Imitation Ding porcelains (period 2); square: ordinary Cizhou porcelains from periods 1—3 (Northern Song and Jin Dynasties); cross: ordinary Cizhou porcelains from period 4 (Yuan Dynasty).

Chinese ceramics.

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