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Determination of 16 heavy metal elements and 16 rare earth elements in Ya'an tibet tea by ICP-MS

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Abstract. The contents of 16 heavy metal elements such as As,Be,Co,Cr,Cu,Ga,Li,Ni, Sr,V,Zn,Ag,Cd,Cs,Bi, Pb and 16 rare earth elements such as Sc, Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm,Yb, Lu were analyzed, simultaneously and effectively by inductively coupled plasma-mass spectrometry (ICP-MS) method with high pressure digestion and Rh, Re as the internal standard elements. The linear correlation coefficient of each element standard curve was 0.9919- 0.9999; the detection limit of each element is 0.0003 ug/L -0.2164 ug/L; the daily and daytime deviation of each element was lower; the average recoveries were between 93.39% -110.99%, RSD was less than 7.49%. The results showed that the average content of Zn was the highest to be (32.833±0.993) mg/kg, followed by Cu, Sr and Ni, which were significantly higher than those of other 28 elements. The total amount of rare earth in Ya'an was 0.588 mg/kg -3.161 mg/kg, The average content of Ce was the highest, followed by La, Sc, Nd and Y, the total amount of rare earth total 83.16%-86.15%, the average content of Lu was the lowest. The proportion of each element in the soil, different tea varieties on the absorption and enrichment of various elements, application of fertilizer and pesticide in tea garden resulted in the great differences of each element content.

1. Introduction

Tea contains many kinds of nutritional components, such as tea polyphenols, amino acids and so on. It also contains a variety of trace metal elements. These elements may bring beneficial effects. For example, Zn, Cu, an excess free radical remover in the body, can effectively increase immunity and delay aging [1]. At the same time, they may also threaten the life and health of humans [2]. Excessive intake of heavy metals such as Cd, Cr, Pb, As and Se in diet will increase the risk of cancer to some extent [3]. Rare earth elements in tea enter human body through food chain, and trace rare earth can play a role in health care. However, excessive intake will accumulate in different parts of the body and lead to lesions, and may lead to acute myocardial infarction, leukemia and so on [4-5]. Therefore, human health problems caused by heavy metals and rare earth elements are attracting more and more attention. ICP-MS is a multi-element rapid detection technology. Because of its high sensitivity and simple and effective advantages, it is used for the determination of trace metal elements, especially rare earth elements in tea [6-8]. It is mainly applied to the identification of origin and safety application of tea [9-11]. Ya'an Tibetan Tea is a famous black tea in Sichuan South Road side tea. It uses the processing technology of south side tea and innovates the characteristic tea that is suitable for domestic sale, overseas sale and export tea products according to market demand. In recent years, the upsurge of Tibetan tea drinking has sprang up, and the tea industry of domestic sales has made great progress. At present, there is no systematic study on the safety detection of heavy metals and rare earth elements in Ya'an Tibetan tea. In this experiment, Ya'an Tibetan tea was taken as the research object, with high pressure digestion for sample pretreatment, Rh and Re as internal standard elements. A rapid and effective method was established by ICP-MS, at the same time, 16 kinds of heavy metal elements such as As, Be, Co, Cr, Cu, Ga, Li, Ni, Sr, V, Zn, Ag, Cd, Cs, Bi, Pb etc. and 16 rare earth elements such as Sc, Y, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu etc. were quickly and effectively measured. It provides technical support for Ya'an Tibetan tea quality and quality detection and comprehensive benefit and its sustainable development.

2. Materials and Methods

2.1. Materials and reagents
Ya'an Tibetan tea was provided by the main tea producing enterprises in Ya'an, Sichuan Province, all of which were spring tea. The sample was fully grated and spare.

16 kinds of heavy metal storage solution and mass spectrum tuning solution were purchased from PerkinElmer company of the United States. Rare earth and In, Re and Rh internal standard storage solutions are purchased from the national nonferrous metals and electronic materials analysis and testing center. Nitric acid was purchased from Merck company in Germany. The experiment water was ultra pure water. The glassware used was soaked in 20% nitric acid solution for more than 24 h and rinsed clean with ultra pure water.

2.2. Instrument and equipment
NexION 300Q ICP-MS is from PerkinElmer United States. HPA-S high pressure ashing apparatus is from Anton Paar company in the Austria. ALC-110.4 electronic balance is from Sartorius company in the German. Arium Comfort water purifier is from Sartorius company in the German.

2.3. Experimental method

2.3.1. Sample digestion
The dried tea powder was accurately weighed 0.35g in the quartz digestion tank, adding 3 mL of concentrated nitric acid. After, the seal was put into the high pressure ashing apparatus. Dissolve according to the following procedures: The temperature was first heated to 70 degrees, and raised temperature to 120℃within 20 min, then warmed up to 180℃and held at 180℃ for 60 minutes. After cooling, the treatment solution was taken out of the digestion tank, and the volume was adjusted to 25 mL volumetric flask with ultrapure water, shaken for use, and after appropriate dilution, it was measured on the machine. At the same time, a blank control was made.

2.3.2. ICP-MS working parameters
By using the standard detection mode, the performance state of the instrument and the parameters of the instrument are optimized by inhaling mass spectrometry tuning liquid. The sensitivity, oxide, double charge and resolution of the instrument meet the requirements of measurement. The specific parameters were as follows: ICP RF power: 1050W, Atomizer flow: 0.89 L/min, Auxiliary gas flow: 1.2 L/min, Plasma gas flow: 16 L/min, Atomizer: Concentric Nebulizer, Scanning times: 20, Number of readings: 1, Repetitions: 3, Detector: Double mode, Acquisition mode: Jump peak, Detection method: automatic, Integral time: 36s, Be＞3000 cps, Mg＞20000 cps, In＞50000 cps, U＞40000 cps, CeO/Ce≤ 3%, Mass number 220 background≤ 5%.

2.3.3. Standard solution and internal standard solution
The 1% nitric acid solution was used to dilute the mixed storage liquid of 10 ug/mL and 16 kinds of the heavy metal mixed standard storage liquid such as As, Be, Co, Cr, Cu, Ga, Li, Ni, Sr, V, Zn, Ag, Cd, Cs, Bi, Pb, etc. step by step to the standard solution of different gradient. Among rare earth

solution: 0, 0.5, 1.0, 2.0, 5.0, 10.0 ug/L. The other 16 kinds of heavy metal mixed standard solutions: 0, 1.0, 2.0, 5.0, 10.0, 20.0 ug/L. The 100 ug/mL In, Re and Rh mixed internal standard storage solution was diluted to 1 ug/L by 1% nitric acid solution.

2.4. Data analysis

All the results were expressed in mean value of ± SD (n=3), and Excel 2003 was used to analyze the significance of variance.

3. Results and Analysis

3.1. The selection of the internal standard elements and the number of the elements to be measured

This study involved 32 different metal elements, so on the basis of the satisfactory results of the internal standard stability test, the mass fraction of the elements with large abundance, high sensitivity and small disturbance should be selected. Comparing the three internal standard tests of In, Re and Rh, in this experiment, $Rh^{102.905}$ was finally chosen as the internal standard of $Sc^{44.956}$、$Y^{88.905}$、$As^{74.922}$、$Be^{9.012}$、$Co^{58.933}$、$Cr^{51.941}$、$Cu^{62.930}$、$Ga^{68.926}$、$Li^{7.016}$、$Ni^{57.935}$、$Sr^{87.906}$、$V^{50.944}$、$Zn^{63.929}$、$La^{138.906}$、$Ce^{139.905}$、$Pr^{140.907}$、$Nd^{145.913}$、$Sm^{146.915}$、$Eu^{152.929}$、$Gd^{156.934}$、$Tb^{158.925}$、$Dy^{162.925}$、$Ag^{106.905}$、$Cd^{111.903}$、$Cs^{132.905}$. $Re^{184.953}$ was chosen as the internal standard of $Ho^{164.930}$、$Er^{165.930}$、$Tm^{168.934}$、$Yb^{171.941}$、$Lu^{174.941}$、$Bi^{208.980}$、$Pb^{207.977}$.

3.2. Selection of sample digestion methods

Samples preparation is the key to affect the accuracy of element content. At present, most of the metal elements digestion use microwave digestion[12]. But the consumption of reagents is large. When completing the resolution, it is necessary to remove the acid from the digestion solution to avoid the interference of measuring signals and instrument damage to ICP-MS. In this experiment, tea standard substance GBW 10016 was used as sample for microwave digestion and high pressure digestion respectively. The results showed that the content of 32 metal elements, recovery rate, etc. can meet the simultaneous determination of 32 elements. However, microwave digestion can reduce reagent consumption, reduce environmental pollution and do not need to run sour. Therefore, high pressure digestion method was adopted.

3.3. The standard curve, linear range and detection limit of each element

The standard curve is obtained by taking the element concentration as the abscissa and the ratio of the response value of the measured element to the internal standard element as ordinate. The detection limit of the method is 3 times the standard deviation of blank value of 11 samples [13].

Table 1. Standard curves, linear range and limit of detection of 32 elements.

element	regression equation	r	LOD/ (ug·L^{-1})	element	regression equation	r	LOD/ (ug·L^{-1})
Sc	y = 0.8428 x - 0.0374	0.9999	0.0354	Nd	y =0.2546 x + 0.0025	0.9999	0.0018
Y	y = 1.2959 x +0.0349	0.9999	0.0017	Sm	y = 0.2174 x +0.0038	0.9999	0.0043
As	y =0.0734 x + 0.0064	0.9999	0.0094	Eu	y = 0.7596 x +0.0203	0.9999	0.0006
Be	y = 0.0743 x +0.0008	0.9999	0.0023	Gd	y = 0.2719 x +0.0012	0.9999	0.0009
Co	y = 0.6732 x +0.0372	0.9999	0.0010	Tb	y = 1.3664x + 0.0400	0.9999	0.0006
Cr	y = 0.5384x + 0.0310	0.9999	0.0762	Dy	y = 0.3401 x +0.0063	0.9999	0.0006
Cu	y = 0.3081x + 0.0624	0.9999	0.0196	Ag	y = 0.4650 x +0.0359	0.9999	0.0113
Ga	y = 0.5815 x +0.0460	0.9999	0.0100	Cd	y = 0.1945x + 0.0119	0.9999	0.0006
Li	y = 0.3130 x +0.0220	0.9999	0.0100	Cs	y = 1.1620 x +0.1036	0.9999	0.0013
Ni	y = 0.3590 x +0.0344	0.9999	0.1356	Ho	y = 3.3012 x +0.6304	0.9990	0.0007
Sr	y = 1.0534 x +0.0945	0.9999	0.0039	Er	y = 1.0833x + 0.2101	0.9991	0.0010
V	y = 0.6380 x +0.0222	0.9999	0.0060	Tm	y = 3.2495 x +0.5665	0.9992	0.0003

Zn	y = 0.1495x + 0.4507	0.9916	0.2164	Yb	y = 0.7195x + 0.1299	0.9992	0.0048
La	y = 1.2685 x +0.0100	0.9999	0.0009	Lu	y = 3.0633 x +0.4995	0.9992	0.0009
Ce	y = 1.1841 x +0.0508	0.9999	0.0008	Bi	y = 1.6176 x +0.5646	0.9992	0.0011
Pr	y = 1.4612 x +0.0191	0.9999	0.0004	Pb	y = 1.0410 x +1.0453	0.9999	0.1122

As can be seen from Table 1, the 32-element standard curve had a good linear relationship and the correlation coefficients were mostly 0.9999. The detection limit of the 32 elements was very low, from 0.0003ug/L to 0.2164ug/L. Zn had the highest detection limit, and the detection limit of Ni and Tm was the lowest. The correlation coefficient and detection limit of each element can meet the requirements of quantitative analysis.

3.4. Repeatability, stability and recovery of the method

Within-day precision is measured the same tea reference material which is GBW10016 GSB-7 five times in a row on the same day. The inter-day precision is measured on the same sample for 3 consecutive days, measured 3 times a day, and averaged. The results showed that the RSD of within-day precision and inter-day precision were 0.12~3.85% and 1.21%~6.65% respectively. The within-day precision RSD of each element was less than the inter-day precision (Table 2). Each element had a low within-day and inter-day deviation, and the within-day deviation was less than the inter-day deviation. It showed that the stability and reproducibility of the method was good.

The recovery rate was the tea standard substance GBW10016 GSB-7 with known content, adding 3 different concentration of standard liquid. When completing the resolution, the sample was determined and the recovery rate of each component was calculated. The results showed that the recovery rate was between from 93.39% to 110.99%. The recovery rate of RSD was less than 7.49% (Table 2).

Table 2. Precision, reproducibility, and recovery of ICP-MS method

element	within-day precision/%	inter-day/%	recovery /%	recovery RSD/%	element	within-day precision/%	inter-day /%	recovery /%	recovery RSD/%
Sc	0.68	3.78	105.83	2.48	Nd	1.80	6.62	105.46	1.08
Y	0.85	2.46	107.61	5.71	Sm	1.12	5.85	110.99	1.58
As	1.02	4.26	101.43	4.52	Eu	1.52	3.30	108.35	6.37
Be	1.49	2.34	95.37	0.55	Gd	1.54	1.21	107.91	7.30
Co	1.09	1.79	104.85	3.21	Tb	0.61	5.00	103.19	3.12
Cr	0.87	6.65	93.49	5.25	Dy	1.07	2.81	104.67	1.38
Cu	0.25	3.75	99.59	4.14	Ag	3.85	3.51	103.88	4.08
Ga	1.26	2.54	107.63	1.41	Cd	0.51	3.61	93.39	7.49
Li	1.68	3.77	100.17	4.56	Cs	0.49	4.44	101.95	1.60
Ni	0.13	6.22	97.24	4.01	Ho	0.57	3.89	104.24	3.93
Sr	0.84	5.84	96.80	5.41	Er	1.02	3.36	104.88	2.27
V	0.75	3.31	104.55	6.76	Tm	1.17	1.42	105.67	3.27
Zn	0.12	2.78	99.28	3.31	Yb	0.27	2.79	104.65	0.82
La	0.12	3.46	101.51	2.94	Lu	0.24	2.16	102.97	6.12
Ce	0.57	1.61	98.60	5.37	Bi	1.00	5.57	94.57	4.41
Pr	0.36	5.13	103.57	5.59	Pb	0.44	2.33	93.68	4.29

3.5. Simultaneous determination of 32 elements in Ya'an Tibetan tea by ICP-MS

The content of 32 elements in Ya'an Tibetan tea was shown in Table 3. The results showed that the average content of Zn was the highest (32.833±0.993) mg/kg, followed by Cu (16.143±0.658)mg/kg, Sr (12.571±0.659)mg/kg and Ni (9.155±0.210)mg/kg. They were significantly higher than those of the other 28 elements ($P<0.010$). At present, NY/T 288-2012 requires Cu is less than 30 mg/kg. GB 2762-2017 requires that the lead is less than 5 mg/kg, and deletes the requirements for the limit of rare earth. NY 659-2003 requires that the chromium is less than 5 mg/kg. Cadmium is less than 1 mg/kg.

Arsenic is less than 2 mg/kg. It shows that the chromium, cadmium and arsenic in Ya'an Tibetan tea have not exceeded the standard. There is no limit on other elements in the current tea standards.

The total amount of rare earth elements in Ya'an Tibetan tea was from 0.588 mg/kg to3.161 mg/kg, with an average of (1.699±0.016) mg/kg. Rare earth elements included 16 elements: light rare earth (La, Ce, Pr, Nd, Sm, Eu, Gd) and heavy rare earth (Tb, Dy, Ho, Er, Tm, Yb, Lu, Y, Sc). As shown in Table 3, the total amount of light rare earth elements Ce, Eu, La, Nd, Pr, Sm accounted for 56.77~70.07% of the total content of 16 rare earth elements, which was significantly higher than the total amount of heavy rare earth elements Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Sc and Y ($P < 0.01$). This conclusion was consistent with the previous research [13]. The average content of Ce in Ya'an Tibetan tea was the highest among all rare earth elements (0.546±0.012) mg/kg, followed by La (0.257±0.004) mg/kg, Sc (0.248±0.004) mg/kg, Nd (0.195±0.008) mg/kg and Y (0.193±0.003)mg/kg. The total amount of Ce, La, Sc, Nd, and Y accounted for 83.16% ~ 86.15% of the total amount of rare earths. The average content of Lu (0.002±0.000) mg/kg was the lowest. Sc and Y were heavy rare earth elements, whose average content was significantly higher than t Tea can be used for the treatment and prevention of many diseases. Theoretically, tea should not be contaminated by heavy metals. However, in fact, these metal elements can be accumulated in the acidic soil to absorb tea leaves. The average content of Zn in 6 Ya'an Tibetan tea produced by main producing enterprises of Ya'an Tibetan tea was (32.833±0.993) mg/kg. The average content of Cu, Sr and Ni was over 9 mg/kg. The total amount of 2 rare earth elements was higher than 3 mg/kg. Why was there a high content of heavy metals and rare earth elements in Ya'an Tibetan tea? First, there were high content of heavy metals and rare earth elements in soil. Second, the absorption and enrichment of heavy metals and rare earth elements in different tea varieties were different [14]. Third, the cultivation and management of tea plantations were also inseparable, such as foliar fertilizers containing heavy metals and rare earths, compound fertilizers and spraying rare earth containing pesticides [15]. Fourth, the accumulation of rare earth elements in Ya'an Tibetan tea can be caused by the hygrothermal effect and the participation of microorganisms [16].hat of some light rare earth elements ($P < 0.01$), such as Pr, Sm and Eu.

Table 3. Contents of 32 elements in Ya'an Tibet tea (mg/kg)

element	1	2	3	4	5	6	average content
Sc	0.363±0.029	0.174±0.025	0.617±0.014	0.137±0.010	0.160±0.016	0.484±0.012	0.248±0.004
Y	0.337±0.005	0.110±0.003	0.084±0.003	0.074±0.002	0.101±0.002	0.450±0.009	0.193±0.003
As	0.131±0.007	0.078±0.003	0.075±0.002	0.060±0.001	0.118±0.007	0.377±0.023	0.140±0.001
Be	0.047±0.004	0.018±0.002	0.012±0.001	0.009±0.000	0.017±0.002	0.060±0.001	0.027±0.000
Co	0.621±0.023	0.610±0.006	0.567±0.014	0.398±0.010	0.588±0.018	0.686±0.004	0.578±0.011
Cr	2.381±0.086	0.988±0.021	1.577±0.072	0.805±0.048	1.180±0.042	3.724±0.083	1.776±0.059
Cu	13.096±0.718	16.968±0.974	15.967±0.822	16.251±0.382	17.894±0.533	16.684±0.517	16.143±0.658
Ga	1.009±0.052	0.513±0.012	0.584±0.011	0.300±0.011	0.502±0.011	1.440±0.035	0.725±0.022
Li	0.461±0.039	0.307±0.030	0.259±0.020	0.310±0.027	0.262±0.023	0.661±0.032	0.377±0.005
Ni	7.718±0.536	9.169±0.425	11.191±0.243	8.494±0.359	9.880±0.372	8.476±0.399	9.155±0.210
Sr	16.483±0.299	9.653±1.136	11.008±0.766	4.755±0.417	8.755±0.402	24.771±0.932	12.571±0.659
V	0.631±0.010	0.263±0.007	0.318±0.005	0.218±0.006	0.284±0.003	1.000±0.016	0.452±0.004
Zn	22.357±0.636	30.139±0.686	36.220±2.269	46.158±1.904	35.376±1.022	26.745±0.714	32.833±0.993
La	0.483±0.027	0.131±0.015	0.113±0.011	0.079±0.009	0.120±0.015	0.618±0.021	0.257±0.004
Ce	1.158±0.025	0.234±0.027	0.191±0.019	0.153±0.012	0.218±0.009	1.323±0.028	0.546±0.012
Pr	0.097±0.002	0.028±0.002	0.021±0.003	0.018±0.001	0.023±0.005	0.144±0.020	0.055±0.002
Nd	0.362±0.013	0.103±0.008	0.079±0.010	0.063±0.002	0.093±0.008	0.468±0.036	0.195±0.008
Sm	0.070±0.003	0.021±0.001	0.017±0.001	0.012±0.002	0.020±0.001	0.096±0.011	0.039±0.003
Eu	0.046±0.002	0.018±0.000	0.019±0.000	0.010±0.000	0.018±0.001	0.057±0.001	0.028±0.000
Gd	0.090±0.006	0.025±0.001	0.019±0.002	0.015±0.001	0.023±0.001	0.138±0.027	0.052±0.005
Tb	0.012±0.001	0.004±0.000	0.003±0.000	0.002±0.000	0.004±0.000	0.017±0.000	0.007±0.000
Dy	0.059±0.002	0.018±0.000	0.013±0.000	0.011±0.001	0.016±0.000	0.083±0.006	0.033±0.001
Ag	—	0.027±0.007	0.067±0.008	0.016±0.004	0.004±0.001	0.018±0.007	0.021±0.004
Cd	0.063±0.005	0.080±0.009	0.063±0.005	0.088±0.007	0.067±0.009	0.147±0.020	0.085±0.008

Cs	0.290±0.004	0.217±0.001	0.115±0.004	0.153±0.002	0.199±0.005	0.382±0.001	0.226±0.001
Ho	0.011±0.000	0.003±0.000	0.004±0.001	0.002±0.000	0.003±0.000	0.016±0.000	0.007±0.000
Er	0.035±0.002	0.010±0.000	0.008±0.001	0.006±0.000	0.010±0.001	0.047±0.002	0.019±0.001
Tm	0.004±0.000	0.001±0.000	0.001±0.000	0.001±0.000	0.002±0.001	0.006±0.000	0.003±0.000
Yb	0.029±0.000	0.008±0.000	0.006±0.001	0.005±0.000	0.008±0.000	0.040±0.001	0.016±0.000
Lu	0.004±0.000	0.001±0.000	0.001±0.000	0.001±0.000	0.001±0.000	0.005±0.000	0.002±0.000
Bi	0.027±0.001	0.007±0.001	0.010±0.000	0.007±0.001	0.009±0.000	0.060±0.004	0.020±0.001
Pb	1.485±0.042	0.650±0.027	0.546±0.010	0.829±0.042	0.591±0.029	4.200±0.082	1.383±0.011
Totle	3.161±0.101	0.890±0.084	0.746±0.003	0.588±0.018	0.819±0.054	3.990±0.038	1.699±0.016

4. Conclusion

In this experiment, Rh and Re were internal standard elements and the samples were digested by high pressure digestion. An ICP-MS method was established to simultaneously detect the contents of 16 heavy metals and 16 rare earth elements in Ya’an Tibetan tea. This method is simple, reproducible, stable and high recovery rate. This method is suitable for rapid and effective detection of multi element pollutants in tea, providing technical support for quality quality testing and comprehensive benefits of Ya'an Tibetan tea, which is of great significance for the sustainable development of Ya'an Tibetan tea industry.

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