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PO-CON1420E

Determination of major and minor elements in the marine sediments of manganese crusts by ICP-OES

Pittcon 2014 2280-9P

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Introduction

Develop the marine resources, protect the maritime environment, maintain the national maritime rights and interests are the current trend in the world. Certainly, resources and environment are inseparable. However, even if you develop the marine resources or protect the maritime environment, we should know its chemical composition firstly, so analytic technology is always essential to the fundamental studies. Marine sediments occurred due to a variety of marine deposition.

Determination of constant or trace elements in the marine sediments is essential to marine science, environmental protection and geological chemistry. Here we introduce analysis of trace constituents in manganese crust using the multi-type ICPE-9000 ICP emission spectrometer. ICP emission spectrometry is highly sensitive and features a wide dynamic range, thereby enabling simultaneous analysis of these principle ingredients as well as toxic trace constituents.

Experimental

Analysis by ICP-AES

Sample preparation procedure :

(1) After heating the GBW07296 sample dry in the bake oven (about 50 °C), transferred it to the agate mortar and grinded into 200 mesh.

(2) After drying the crushed sample at 105 °C for 3 hours, cooled to room temperature, weigh about the 50.00 mg samples accurately in the tanks, added 1.50 mL HNO_3 and 1.50 mL HF, stamped and sealed, then heated in the bake oven about 195 °C for more than 48h.

(3) Until the tanks get cool, and perform heat-decomposition by heating it on the hot plate until the sample change into the wet salt, then add 1mL HNO_3 (remove the remnants of HF).
Eventually added 3 mL of high purity HNO_3, stamped and sealed, heating it in the bake oven for 24h in order to extraction of the samples completely.

(4) After cooling, transferred the solution to the PET (polyester) volumetric flask, bring volume to 25.00g using pure water, and use this as the analytical sample.

ICP emission spectrometry (ICP-AES) is highly sensitive allows simultaneously analysis of multiple elements, and features a wide dynamic range. It can therefore be used for efficient analysis not only of the principle high-concentration constituents, but of trace elements as well. We introduce here the use of the multi-type ICPE-9000 for quantitative of the content of Al, Ba, Ce, Co, Cu, K, Na, La, Mo, Ni, P, Pb, Sr, Ti, V, Y, Zn and Zr in the manganese crusts.

Figure 1. Shimadzu Multitype ICP Emission Spectrometer

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Excellence features of ICPE-9000

(1) Adopts a vacuum type spectrometer, not offered by other companies

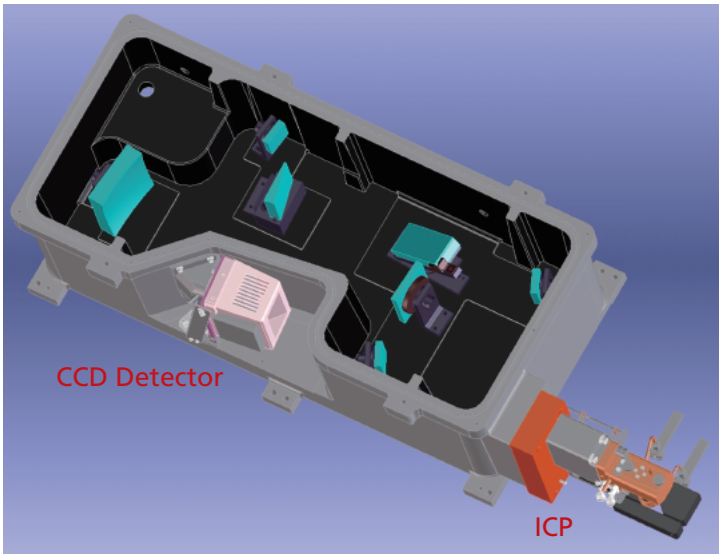
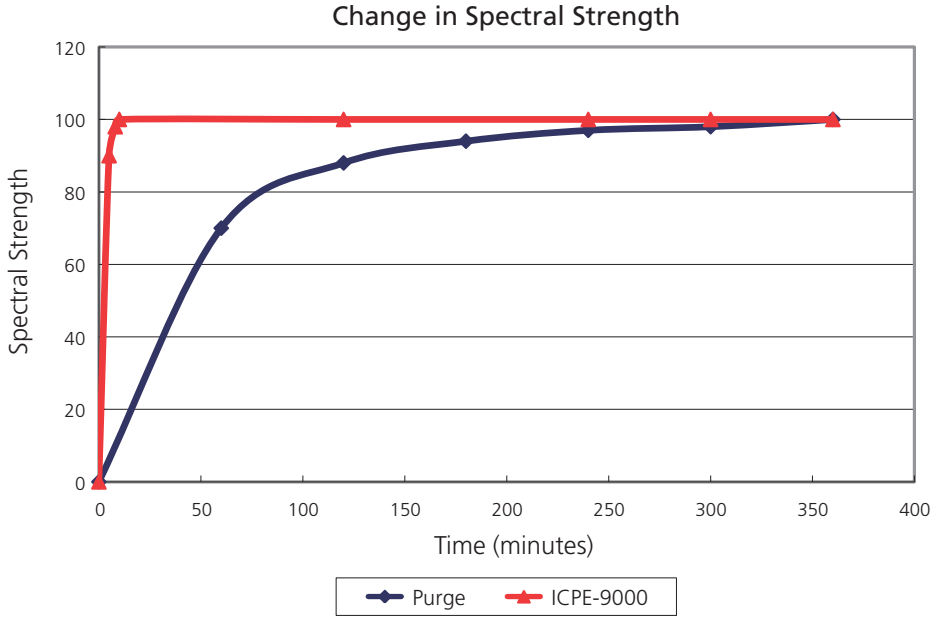


Figure 2. vacuum type spectrometer of ICPE-9000

With the vacuum spectrometer, absorption is eliminated by maintaining a vacuum of approximately 15Pa. There is no impact on spectrometer temperature, enabling stable measurements. As purge gas is not required, running costs are low.



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(2) Low running costs

The Shimadzu Corporation proprietary mini torch has been adopted.

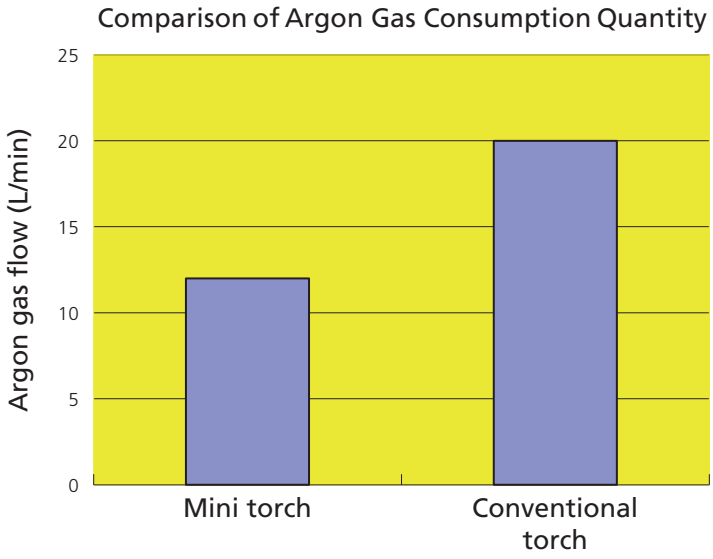

Table 1 ICP-AES Analysis Conditions

RF Power	1.2 (Kw)
Plasma gas (Ar)	10 (L/min)
Auxiliary (Ar)	0.7 (L/min)
Carrier gas (Ar)	0.6 (L/min)
Nebulizer	Coaxial Nebulizer
Spray chamber	Cyclone Chamber
Observation direction	Axial/Radial

Analysis and Results

The measurement results for the 18 elements obtained using ICP-AES are shown in Table2. The quantitation values matched the certified value of GBW07296 (Provided by the first institute of oceanography, SOA.). It is clear that 18 elements in the manganese crusts can be accurately analyzed with high sensitivity using the ICPE-9000.

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Table 2 Quantitation Results and Detection Limit

Element	Detection Limit (mg/L)	GBW07296 Certified Value	GBW07296 Quantitation Results	Unit	RSD (%)
Al_2O_3	0.03	4.7±0.3	4.40	%	0.54
Ba	0.0004	0.24±0.02	0.226	%	0.7
Ce	0.002	249±21	253	μg/g	0.48
Co	0.0005	0.17±0.01	0.16	%	1.25
Cu	0.001	1.36±0.04	1.36	%	1.17
K_2O	0.02	1.14±0.05	1.09	%	1.95
La	0.0003	96±10	91.3	μg/g	0.68
Mo	0.0007	622±37	590	μg/g	0.62
Na_2O	0.05	3.03±0.14	2.95	%	0.54
Ni	0.004	1.55±0.07	1.55	%	1.18
P_2O_5	0.03	0.37±0.06	0.33	%	2.04
Pb	0.004	328±33	308	μg/g	0.43
Sr	0.0001	561±62	540	μg/g	0.77
TiO_2	0.001	0.54±0.04	0.50	%	0.70
V	0.0003	442±51	414	μg/g	0.07
Y	0.001	84±3	81.60	μg/g	0.20
Zn	0.004	0.16±0.01	0.156	%	0.76
Zr	0.0001	256±31	241	μg/g	0.33

Conclusion

This paper using the HF-HCl-HNO_3 system with high-pressure closed digestion for samples pre-treatment, and utilizing ICP-AES for determination the 18 multielements in the manganese crusts. The experimental results show that, good linear relationship with linear correlation coefficient r>0.9998. The detection limits of these 18 elements are low, the precision of RSD less than 2.0% (n=6), The quantitation values matched the certified value of manganese nodule GBW07296. This method can be widely used in geological samples.

First Edition: March, 2014

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