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Comparison of USEPA Digestion Methods for Trace Metal Analysis Using SRM

Mi-Young Shin, Hyeon Yoon, Youn-Tae Kim*, Cheol-Ho Yoon, Nam-Chil Woo*

*Korea Basic Science Institute, *Dep. of Earth System Sciences, Yonsei University (popo482@kbsi.re.kr)*

Abstract

The importance of a proper sample preparation and analysis technique has getting attention due to the complicity of toxic elements of interest from environmental samples depend on analytical purposes. It is critical to use proper analytical method to evaluate trace elements concentration in many environmental samples especially for making remediation decisions. Therefore, it is critical to apply a proper sampling and analytical method such as EPA publication SW-846 (Test Methods for Evaluating Solid Waste, Physical/Chemical Methods).

The objective of this study was to compare the USEPA Methods 3050B, modified 3050B, 3051a, and KBSI method (modified EPA 3052 Method) in term of recovery rate of metals. The NIST SRM (Montana soil) was used to compare the extraction and digestion efficiency. After sample has been collected the analysis were achieved by ICP-MS (Elan 6100, Perkin Elmer) as well as ICP-AES (Ultima 2C, JY) for trace elements and major elements.

key word : analysis, toxic elements, EPA SW-846 Test Methods, KBSI Method

1. Introduction

Toxic heavy metals from contaminated site is concern of today due to its lasting effect on land and streams nearby. In order to evaluate the toxicity of heavy metals, development of a substantial protocol is necessary for determining background concentrations of heavy metals from clean land as well as contaminant site.

Several analytical techniques have been developed including USEPA Methods for correct determination of toxic elements in soils. The sample preparation technique are distinguished either a total analysis or a total recoverable analysis method. A total analysis method of metal ions requires more than one day until the complete digestion of sample materials. This method requires the use of strong acid to get a complete dissociation of sample materials and the high temperature thermal decomposition process to enhance the chemical reaction. However, the total analysis method suffers not only the loss of elements of interest such as arsenics but also duration of reaction time as well

as incomplete recoveries toward complete digestion. Compare to this, a alternative method, total recoverable digestion, have become a common method for sample digestion. The total recoverable digestion method in environmental samples especially for the monitoring of the mobile toxic elements in contaminated areas. It is very important to evaluate a proper sample preparation method depend on the purposes of analysis.

In this study, we present a comparison of four sample preparation methods, three USEPA Methods including a modified one (USEPA Method 3050B, 3050B-modified, 3051a) and KBSI Digestion Method, for determining concentration of environmentally hazardous elements of interest from the soil SRM sample.

2. Materials and Method

Materials

NIST SRM 2710 (Montana Soil) was used in this study (NIST 2003). Detailed chemical composition of this soil is presented in table 1. Standard stock solutions of 1000mg/L of each elements of interest is obtained from Spex Industries (Edison, NJ). All reagent grade chemicals used during this study were obtained from Merck (GR garde).

Sample Digestion Methods and Instrumentation

USEPA Method 3050B for all elements analysis, 3050B for other elements such as Sb, Ba, Pb, and As, 3051a, and KBSI (Modifed from 3052, Korea Basic Science Institute) digestion Method were used for the determination of elements of interest in SRM samples. Also samples were treated with aqua regia for complete metals digestion.

The schematic diagram of two 3050B Methods were presented in figure 1 and 2.

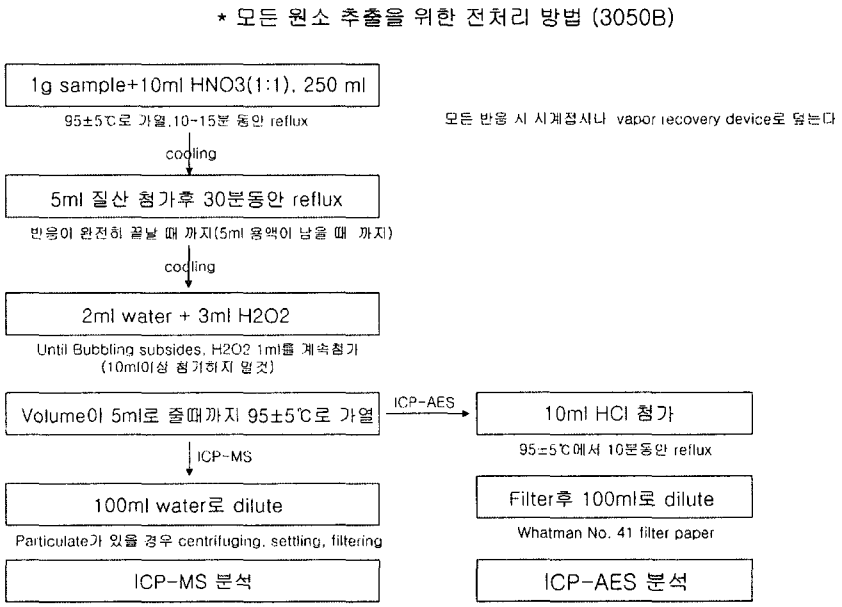

Figure 1. USEPA Method 3050B for all elements analysis.

3. Result and Discussions

After the samples were prepared for elemental analysis by ICP-AES (Ultima 2C, Jobin Yvon, France), instrumental analysis was pursued and the results were evaluate according to the efficiencies of each method for best correct determination of elements of interests.

UESEPA Method 3050B-totaland 3050B-selective Methods are satisfactory for all elements except for As, Ba, Mg from Montana Soil. This Method is widely accepted one but still suffers low recovery of Al, Cr, Fe, Mg, Mn, and Zn. However, Method 3051a show more enhance results in Ag, Al, and Fe recoveries. Compare to this KBSI Method is the one modifed from USEPA Method 3052 by adding HF-$HClO_4$-HNO_3 step during total digestion of soils, ceramics, and silicates. As it appeared in Table 1, KBSI Method show almost complete recoveries of all elements appeared in certification of NIST SRM (Montana Soil).

As we early mentioned, comparing total decomposition method (KBSI Method modified from USEPA Method 3052) to total recoverable digestion method (3050B, 3051a) show great differences in Al, Ca, Cr, Fe, Mn, Pb, and Zn. But no significant differences was found in Mo, Cd, Ni, and Ag. This difference is explained by the mineralogical configuration of SRM (Montana Soil) in which most metal ions were strongly bounded to the silicate mineral.

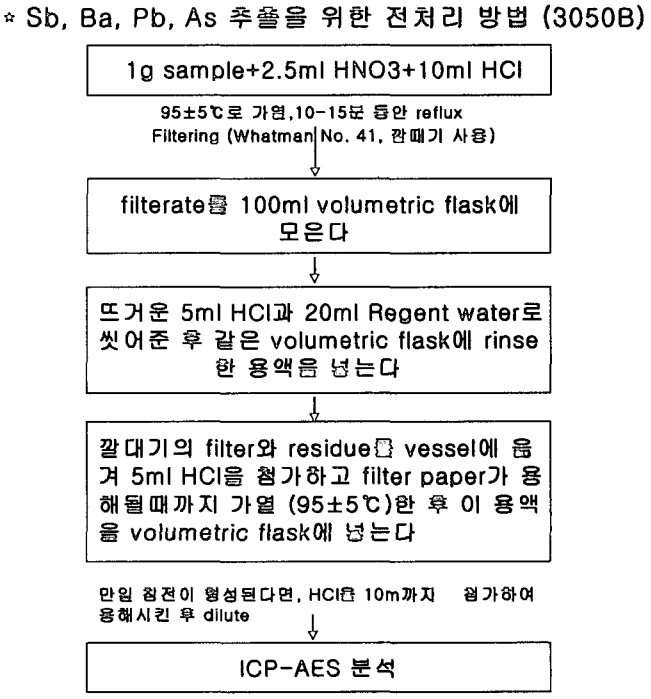

Figure 2. USEPA Method 3050B for Sb, Ba, Pb, and As analysis.

Table 1. Concentration of 17 elements in NIST SRM 2710 digested by USEPA Methods 3050B, 3051a, Aqua Regia, and KBSI Method (ppm).

	3051a	3050B-total	3050B-Selective	Aua Regia	KBSI
Al	21032	18850	14753	26699	70990
As	627	611	603	635	633
Ba	227	232	220	258	791
Ca	4305	4212	3948	4924	13415
Cr	< 5ppm	< 5ppm	< 5ppm	< 5ppm	67
Cu	3172	3085	3079	3139	3394
Fe	31546	28985	27575	33005	36232
Mg	6096	5632	5021	6735	9477
Mn	9110	8727	8548	9147	11329
Pb	5392	5337	5278	5413	5677
Zn	7128	6877	6603	7079	7827
Mo	5	11	15	10	12
P	959	903	874	935	971
Cd	18	18	17	17	16
Ni	5	8	6	9	9
Ag	23	20	17	17	11

4. References

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