

Application of Microwave digestion in determination of metal elements in solid waste

1. Introduction

Solid waste cause high risk of pollution to soil, surface and ground water and even to the air. The determination of harmful elements inside solid waste plays an important role in environmental protection. Because of the impact of environmental safety to human health, chemical analysis of heavy metal amount in solid waste is compulsory by law in most countries. Heavy metal analysis, e.g. for toxic heavy metals as lead, chromium, cobalt, vanadium etc. are routine analytical tasks in environmental and government testing lab. Here, we present a microwave digestion method which follows local testing standard HJ 766-2015 < Solid waste-determination of metals inductively coupled plasma mass spectrometry (ICP-MS) > to prepare solid waste samples for ICP-MS analysis.

2. Instrument and reagent

Instrument:

The digestions were carried out with TOPEX+ microwave digestion system and GT-240 high pressure digestion rotor. The determination of the heavy metal was conducted by ICP-MS.



TOPEX+ microwave digestion system



GT-240 rotor



G-240 hot block

Reagent:

HNO_3 (GR) ; HCl (GR) ; HF (GR) ; H_2O_2 (GR)

Sample:

GSS-9 standard certified quality control sample

GSS-29 standard certified quality control sample

3. Method

1. Weigh 0.1 g standard certified quality control sample to the vessel.
2. Add HNO_3 , HCl , HF and H_2O_2 to the sample then swirl to mix them thoroughly.
3. Add same amount of acid into the sample cup as sample blank, then seal the vessel.
4. Set the microwave digestion program as shown in the following table:

Table1: microwave digestion program

step	Setting temperature (°C)	Ramp time (min)	Temperature holding (min)
1	120	10	2
2	180	8	2
3	200	6	30

5. Take the vessels out of the cavity when the temperature falls under 60 °C.
6. Open the vessels and place them on the hot block to evaporate acid at 150 °C.
7. Dilute the sample to 50 mL with deionized water when the temperature of the sample cools to room temperature.
8. The final solutions were tested by ICP-MS according to HJ 766-2015 < Solid waste-determination of metals inductively coupled plasma mass spectrometry (ICP-MS) >

4. Result

Table2: ICP-MS result for certified soil sample n = 3

Sample	Weight (g)	Found value mg/kg						
		V	Cr	Co	Ni	Cu	Zn	Pb
GSS-9-1	0.1043	96.32	73.30	14.02	35.83	26.20	61.70	23.32
GSS-9-2	0.1054	95.94	74.68	14.04	35.86	26.57	60.24	23.47
GSS-9-3	0.1051	96.43	74.92	13.99	35.74	26.22	59.83	23.29
Certified value		90±12	75±5	14±2	33±3	25±3	61±5	25±3
GSS-29-2	0.1057	106.01	79.89	16.29	39.13	36.50	93.34	32.19
GSS-29-3	0.103	107.99	80.06	16.54	39.46	35.98	98.27	31.72
GSS-29-4	0.1045	103.36	79.29	16.00	38.78	36.21	94.53	32.28
Certified value		105±3	80±5	16±0.6	38±2	35±2	96±4	32±3

As shown in the result, the method presents good accuracy and recovery in the determination of heavy metal inside solid waste.

5. Conclusion

Preekem's TOPEX+ microwave digestion system coupled with GT-240 rotor can digest solid waste standard samples. The result is a proof for an analytically accurate sample preparation step, which provided the basis for interference free ICP-MS measurement. With the advanced full vessel IR temperature monitor system and pressure control unit, TOPEX+ can ensure the safe and precise sample digestion process during the experiment.