



## High-throughput Acid Digestion or Leaching of Soil Samples With Rotor 41HVT56

Laboratories performing environmental trace analysis of toxicologically and environmentally relevant elements have an ever increasing demand for robust and easy-to-use acid digestion or leaching of soil, sludge and fly ash samples.

With Rotor 41HVT56 the SMART VENT technology was taken to a new level of high throughput for environmental samples.



### 1 Introduction

Routine digestion and leaching of soil samples as well as other environmental samples like sediments, fly ash, sludges etc. are among the most used applications in environmental chemistry. By adding the Rotor 41HVT56 with 41 positions for HVT vessels to the Multiwave PRO platform system the throughput was raised to a new level. The concept of SMART VENT technology allows the HVT56 vessels releasing reaction gases at a controlled pressure. So the robustness of the sample preparation method is increased.

### 2 Instrumentation

The digestions were carried out in HVT56 vessels using Rotor 41HVT56 in Multiwave PRO. The digested solutions were measured with an Agilent 7500ce ICP-MS using He as collision gas to suppress polyatomic interferences



Fig. 1 Rotor 41HVT56

### 3 Experimental

#### 3.1 Samples

The reliability of the method presented was shown by acid leaching and elemental quantification of the certified reference materials "Contaminated Soil" BAM U110 and BAM U112. The different samples were leached in the same run with fully and partially loaded rotor.

#### 3.2 Leaching Procedure

##### 3.2.1 Weighing in the Samples

Sample amounts of 0.5 g were directly weighed into the digestion vessels.

##### 3.2.2 Digesting the Samples

2.5 mL of HNO<sub>3</sub> conc. (65%) and 7.5 mL of HCl conc. (32%) (all analytical grade) were added to the samples. The vessels were closed and placed in the rotor according to the defined loading patterns.

#### NOTICE

To achieve a complete digestion of the samples, hydrofluoric acid may be used to destroy silicates. When working with venting technology the contamination of the cavity and the rotor with HF can be minimized by low-temperature digestion ( $\leq 160^{\circ}\text{C}$ ) or by two-step-digestions.

Table 1: Temperature program for aqua regia leaching of soil samples for Multiwave PRO with Rotor 41HVT56

Step	Temp. [°C]	Time [min]	Fan
Ramp	165	10	1
Hold	165	10	1
Cooling	70	-	3

Maximum Temperature is set to 180°C and the temperature control strategy to AVG (the power introduced is controlled by the average running temperature of all samples).

### 3.2.3 Measuring the Samples

After digestion the samples were transferred into 15 mL centrifuge tubes, the first rinse was merged with the solution and centrifuged together. The supernatant liquid was transferred to another 15 mL vial, the sediment re-suspended in 1 mL of water and centrifuged again, the supernatant was merged with the first and filled to volume. For ICP-MS analysis it was necessary to further dilute the samples 100-fold with DI water.

## 4 Results

The tables below show the certified values in comparison with the measured values.

Table 2: BAM U 110, certified values for leaching with aqua regia and measured values (n=15)

	Certified Values [mg/kg]	Measured Values [mg/kg]	Recovery [%]
<b>Cr</b>	190 ± 9	179 ± 7	94 ± 4
<b>Mn</b>	580 ± 19	531 ± 18	92 ± 3
<b>Co</b>	14.5 ± 0.8	12.9 ± 0.5	89 ± 3
<b>Ni</b>	95.6 ± 4.0	86.2 ± 2.9	90 ± 3
<b>Cu</b>	262 ± 9	230 ± 7	88 ± 3
<b>Zn</b>	990 ± 40	974 ± 33	98 ± 3
<b>As</b>	13 ± 1	13 ± 1	103 ± 5
<b>Cd</b>	7.0 ± 0.4	6.2 ± 0.2	89 ± 3
<b>Hg</b>	49.3 ± 2.9	44.9 ± 1.9	91 ± 4
<b>Pb</b>	185 ± 8	164 ± 6	88 ± 3

Table 3: BAM U 112, certified values for leaching with aqua regia and measured values (n=3)

	Certified Values [mg/kg]	Measured Values [mg/kg]	Recovery [%]
<b>V</b>	12.6 ± 0.8	12.0 ± 0.8	96 ± 6
<b>Cr</b>	78 ± 5	76 ± 5	98 ± 6
<b>Co</b>	3.58 ± 0.23	3.50 ± 0.25	98 ± 7
<b>Ni</b>	9.8 ± 0.5	10.5 ± 0.8	107 ± 8
<b>Cu</b>	74 ± 5	75 ± 8	101 ± 11
<b>Zn</b>	197 ± 11	191 ± 14	97 ± 7
<b>As</b>	10.4 ± 0.4	10.7 ± 0.9	103 ± 8
<b>Cd</b>	3.91 ± 0.24	3.99 ± 0.20	102 ± 7
<b>Hg</b>	16.9 ± 1.4	18.3 ± 2.6	108 ± 16
<b>Pb</b>	195 ± 8	188 ± 9	96 ± 5

The measured values show good agreement with the certified values for a wide range of elements with different concentrations. No analyte losses were observed, even though venting occurred in several vessels during the digestion process.

## 5 Conclusion

The digestion of different soil samples in Multiwave PRO with Rotor 41HVT56 works very well and efficiently. The vessels with SMART VENT technology allow for higher sample amounts and robust operation. Due to the controlled release of reaction gases no losses of volatile elements occur during the digestion procedure.

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