

# Rapid and Efficient Microwave Digestion for Elemental Analysis for Comparative Medicine

2024 Winter Conference on  
Plasma Spectrochemistry  
January 15-20, 2024

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## Introduction

Comparative medicine is built on the ability to use information from one species to understand the same processes in other species. Laboratory rats and mice provide ideal animal models for biomedical research and comparative medicine studies because they have many similarities to humans in terms of anatomy and physiology. Elemental analysis to determine levels of trace elements in these samples can also help assess nutritional status and impact on human health. However, the small size of rats and mice mean that the tissue samples are in milligram quantities. These small sample sizes, coupled with low detection limits, add additional complexity and make sample prep challenging for elemental analysis. In this study, the Discover®SP-D Clinical automated microwave digestion system was used to digest animal tissue. This system can safely operate at temperatures and pressures required to provide a more rapid and efficient digestion.

## Materials and Method

Samples:

- Acid Blank 3 mL HNO<sub>3</sub> + 0.5 mL HCl
- NIST 1577c Bovine Liver
- Adult Rat Kidney

Sample Preparation:

1. Obtain whole rat kidney freshly harvested from an adult male Sprague Dawley rat.
2. Snap freeze kidney and store at -80 °C.
3. Thaw sample in laboratory refrigerator.
4. Homogenize sample using pre-cleaned plastic scoopula.
5. Weigh 0.1 g of sample into a 10 mL quartz vessel with micro stir bar.
6. Add 3 mL HNO<sub>3</sub> + 0.5 mL HCl of trace metal acids to vessel.
7. Cap vessel and place into system or autosampler.

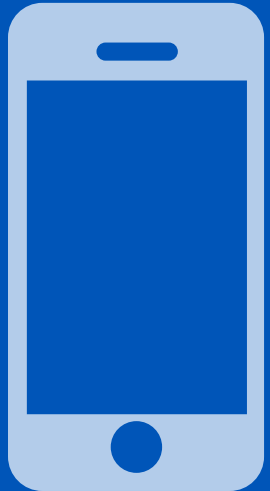


Table 1. Method Parameters

Control Type	Vessel	Pre-stirring (mm:ss)	Stage	Temp (°C)	Ramp Time (mm:ss)	Hold Time (mm:ss)	Pressure (PSI)	Power (W)	Stirring
Ramp to Temp	10 mL Quartz	00:00	1	200	04:00	03:00	400	300	Medium


Table 2. Pressure Stages with Delta Pressure Set to 160 psi

Stage	Pressure SP	Time at SP
1	225	2
2	250	2
3	275	2
4	300	2
5	350	100



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## Analysis

The metals of interest were selected based upon the certified values of the SRMs, as well as industry concerns regarding trace metal contaminants. Samples were analyzed on an Agilent 7850 ICP-MS with conditions listed in **Table 3**. The conditions listed were used to analyze all elements which were analyzed using “H2” and “He” tuning mode. The following elements were used as internal standards: Sc, Ge, Rh, In, Tb, Lu, and Bi. No gas dilution techniques were used during this analysis.

Table 3. Agilent 7850 ICP-MS

Parameter	Value for [He] Mode
RF Power (W)	1550
Sampling Depth (mm)	8
Carrier Gas (L/min)	1.05
Dilution Gas	N/A
Helium Cell Gas (mL/min)	5.0
Energy Discrimination	5.0

## Results

All digests were clear, colorless, and particle-free upon dilution to 50 g with DI water. Acid blanks (**Table 4**) were performed on the trace metals acids to confirm base level values for metals of interest. Background levels were found to be lower than detection limits or negligible. Analysis of the NIST SRM (**Table 5**) demonstrated accurate recovery of the reported values within the expected range. Validation of the standard was used to confirm complete digestion and accurate recoveries of the sample of interest.

Table 4. Average Elemental Recovery (ppb) of Acid Blank (n=3)

	Mg	Ca	Mn	Fe	Cu	Zn	Se	Sr	Mo	Cd	Pb
Acid Blank	0.0350	n.d.	0.0110	0.0483	n.d.	0.0495	0.0083	n.d.	n.d.	n.d.	0.0023

Table 5. Average Elemental Concentrations (ppm) and % Recovery Values for the Certified Elements (n=3)

	Mg	Ca	Mn	Fe	Cu	Zn	Se	Sr	Mo	Cd	Pb
NIST 1577c (Bovine Liver)	562	121	10.2	189	280	170	2.04	0.0930	3.24	0.0938	0.0583
Certified Value	620	131	10.4	198	275	181	2.03	0.0953	3.30	0.0970	0.0628
% Recovery	90.6	92.7	97.3	95.4	102	93.6	100	97.6	98.2	96.7	92.8

Table 6. Average Elemental Concentrations (ppm) of a Single Adult Rat Kidney (n=3)

	Mg	Ca	Mn	Fe	Cu	Zn	Se	Sr	Mo	Cd	Pb
Adult Rat Kidney	182	66.2	0.893	95.0	7.69	22.7	1.33	n.d.	0.247	0.0119	0.00305

## Conclusions

The digestion of a small sample size (approximately 100 mg) of homogenized adult rat kidney was successfully accomplished in under 10 minutes followed by analysis. Comparison of recoveries to reported elements in a NIST SRM demonstrated success of both digestion and analysis. The small sample size was easily handled by the Discover SP-D Clinical automated microwave digestion system and analysis was performed via ICP-MS so that low detection limits could be achieved. The combination of these two technologies is a good fit for comparative medicine, as it allows for both the small sample size as well as the low detection limits required, while still delivering successful results within the expected range.