Calibration Method for Microwave Digestion System

X C Mao¹,², a, T L Xiao¹,², b and J Gu¹, c

¹Shanghai Institute of Measurement and Testing technology, Shanghai, China
²Key laboratory of online testing and control technology of Shanghai, Shanghai, China

maoxc@simt.com.cn; xiaotl@simt.com.cn; guj@simt.com.cn;

Abstract. The article describes the importance of microwave digestion system in many different scientific fields, and proposes a calibration method for lack of calibration specification in China. The method focuses on temperature, pressure and output power. It defines their measuring points, technical requirements. An experiment is carried out to calibrate the output power with its expanded measurement uncertainty. The calibration method can be referenced in daily calibration service of such apparatus.

1. Introduction
Digestion procedure is an important step for sample preparation and analysis. Compared to traditional dry and wet digestion method, microwave digestion is proved to be more accurate, simple and fast. It also provides better heating uniformity, energy conservation and safety [1]. Nowadays, microwave digestion system is widely used in the fields of biology, geology, coal, medicine, food, etc. [2] Studies show that microwave digestion method can be used in many different works, and offer a better performance for them [3-6].

Microwave digestion system generally emits microwave with frequency of 2450MHz. Microwaves can be transmitted through insulators such as vessels or containers that are made of nonpolar molecules. Nearly no energy is absorbed in this progress. When microwave passes through sample, polar molecules in the sample oscillate under the influence of an alternating electric field [7]. Intermolecular collisions and frictions caused by microwave absorption finally produce thermal energy, which makes sample temperature rise [8]. For the oscillation of $2.45 \times 10^9$ times per minute, microwave digestion method, therefore, can accelerate chemical reactions, and improve the uniformity of heat distribution. Figure 1 shows the difference between conventional method and microwave method.

China’s metrology development strategy (2013-2020) focuses much on the research of advanced reference materials, and related technology of separation, purification and conservation for them. It says that improving quality and quantity of reference materials is the key to meet the demands of test and inspection in the fields of biology, food, medicine, metallurgy, etc. As digestion procedure is the first step in analysis, more and more clients now need calibration for microwave digestion systems.

However, there is no current effective standard or verification specification for microwave digestion system in China [9]. GB/T 26814 “Microwave digestion equipment” [10] and GB/T 18800 “Household microwave oven – Methods for measuring performance” [11] are only the related references. As the product standard, the GB/T 26814 pays more attention to product classification, and its test methods are more applicable for manufacturers. GB/T 18800 is mainly aimed at microwave
oven, and the test method is not suitable for daily cycle. Neither of these two standards mentions the output power of microwave digestion system, which is the key parameter of the digestion procedure.

![Figure 1](image)

**Figure 1.** Difference between conventional heating and microwave heating.

2. Calibration method

2.1. Environment requirements

Environment temperature should be 20°C ± 5°C, humidity should be 60%RH ± 20%RH. The machine must be well connected to the ground. Supply voltage of the machine varies within -10%~6% of the rated value. Supply frequency varies within ±5% of the rated value.

Generally, calibration is launched without load. It can be launched in different loading conditions that must be explained. The machine must be placed stable, far away from strong vibration and electromagnetic interference. Around the machine, there must be at least 10 cm space for air circulation, and no other cold or heat source.

2.2. The metrology standards

The metrology standard that will be used are listed in table 1.

| Item         | Name                        | Range          | Margin of error           |
|--------------|-----------------------------|----------------|---------------------------|
| Power        | Precise balance             | (0~4.2)kg      | ±2%                       |
|              | Wireless temperature probe  | (-80~150)°C    | ±0.05°C                   |
|              | Chronograph                 | 0~9h           | ±(5.8 × 10⁻⁶T + 0.01)s    |
|              |                              |                | T: measured time          |
| Temperature  | Metrology well calibrator   | (-40~140)°C    | ±0.1°C                    |
|              |                              | (140~600)°C    | ±0.3°C                    |
| Pressure     | Pressure calibrator         | (0~35)MPa      | ±0.025PS                  |

2.3. Calibration items

2.3.1. Pressure Accuracy. According to the range of pressure measurement system and related national standard, pressure accuracy is determined by details in table 2.

The pressure standard apparatus is connected to the pressure measuring system of the microwave digestion system. Figure 2 shows the connection diagram.
Pressure is increased gradually to each test point in an ascending order according to table 2, then decreased the pressure gradually in a descending order in the same way. The standard and indicated values of pressure are recorded respectively following the calibration methods. It is judged to be qualified when the measurement error meets the requirements in table 2.

**Table 2.** Pressure measuring technical requirements (unit: Mpa).

| Pressure range | Measuring point | Requirements of measurement accuracy |
|----------------|-----------------|--------------------------------------|
| ≤ 5.0          | 0.5             | ±0.1                                 |
|                | 2.5             | ±0.1                                 |
|                | $P - 0.5^a$     | ±0.2                                 |
|                | 0.5             | ±0.1                                 |
| > 5.0 and ≤ 10.0 | 4.5             | ±0.2                                 |
|                | $P - 0.5$       | ±0.5                                 |
|                | 0.5             | ±0.1                                 |
|                | 8.5             | ±0.5                                 |
| > 10.0         | $\frac{1}{2}(10 + P)$ | ±0.6                                 |
|                | $P - 0.5$       | ±0.7                                 |

$^a P$ is the maximum pressure measurement value

2.3.2. Temperature Accuracy. According to the range of temperature measurement system and related national standard, temperature accuracy is determined by details in table 3.

The temperature sensor of microwave digestion system should be inserted into the metrology well calibrator after clean if necessary. Temperature points are chosen according to table 3. After the temperature is stable, the indicated values of Metrology well calibrator and machine can be recorded. It is judged to be qualified when the measurement error meets the requirement in table 3.

2.4. Output Power

A borosilicate glass beaker is employed for calibration. The beaker should have minimum heat capacity so as not to influence the measurement. Steps are as following:

- Environmental temperature is $T_0$. Measure the mass of beaker $m_c$. Stir water with low heat capacity equipment and measure rapidly the initial temperature of water $T_1$. Both $T_0$ and $T_1$ should be less than 25°C.
- Pour water into the beaker and measure the mass of water $m_w$. 

![Connection diagram for pressure measurement](image-url)
• Put the beaker into the microwave equipment, and start the machine. A final water temperature $T_2$ of less than 30°C is preferred. The heating time is noted as $t$.

• Turn off the equipment and measure $T_2$ rapidly.

Output power of microwave digestion system is evaluated according to the equation (1):

$$ p = \frac{4.187 \cdot m_w(T_2 - T_1) + 0.55 \cdot m_c(T_2 - T_0)}{t} \quad (1) $$

### Table 3. Temperature measuring technical requirements (unit: °C).

| Temperature range | Measuring point | Requirements of measurement accuracy |
|-------------------|-----------------|-------------------------------------|
| $\leq 100$        | $T - 10^a$      | $\pm 2$                             |
|                   | 50              | $\pm 2$                             |
| $> 100 \text{ and } \leq 200$ | 100             | $\pm 3$                             |
|                   | $T - 10$        | $\pm 5$                             |
|                   | 50              | $\pm 2$                             |
|                   | 100             | $\pm 3$                             |
| $> 200$           | $\frac{1}{2}(200 + T)$ | $\pm 5$                             |
|                   | $T - 10$        | $\pm 7$                             |

* $T$ is the maximum temperature measurement value

### 3. Experiment for output power

Among these parameters, temperature and pressure are easier to calibrate and influence the digestion effect indirectly. The output power of microwave influences directly on samples. As a result, this chapter proposes the progress for power calibration.

The rated power of the microwave digestion system is 1000 W. The value of power will be accurate to the unit. Related parameters are listed in table 4.

### Table 4. Related parameters for the experiment.

| Symbol | Definition               | Value         |
|--------|--------------------------|---------------|
| $m_w$  | Water mass               | 1000.32 g     |
| $m_c$  | Beaker mass              | 285.38 g      |
| $T_0$  | Environment temperature  | 19.52°C       |
| $T_1$  | Initial water temperature| 10.28°C       |
| $T_2$  | Final water temperature  | 24.36°C       |
| $t$    | Heating time             | 60.3 s        |

Here a wireless temperature probe is employed to measure water temperature. The probe can work in microwave environment. Figure 3 shows the variation of temperature during heating. With the probe, the final temperature can be measured at the highest point of the curve, which means the door is closed and no heat loss occurs. Thus it can offer a more accurate result for power calculation compared with the method in [11].
4. Conclusion
The article describes microwave digestion system and its importance in scientific field. For lack of calibration specification, it proposes calibration method for temperature, pressure and output power of microwave digestion system. It defines measuring points and technical requirements. For the importance of output power, an experiment is carried out to measure the output power of the machine, as well as giving the expanded measurement uncertainty. It shows that with a wireless temperature probe, the measured temperature can reflect more accurately the power of the system. The calibration method can be referenced in calibration of microwave digestion system to guarantee its performance in daily work.

References
[1] Wej R F, Deng Y P and Shi Y H 2018 Microwave digestion technology and its application in analytical chemistry Popular Science & Technology (4) pp. 24-27.
[2] Wan Z 2017 Indication error calibration and uncertainty evaluation of microwave digestion system Industrial Technology Innovation 19(3) pp. 34-36.
[3] Chen C M, Chen M, Leu F C, Hsu S Y, Wang S C, Shu S C and Chen C F 2004 Purification of multi-walled carbon nanotubes by microwave digestion method Diamond and Related Materials vol. 13 pp. 1182-86.
[4] Li X, Dai S F, Zhang W G, Li T, Zheng X and Chen W M 2014 Determination of As and Se in coal and coal combustion products using closed vessel microwave digestion and collision/reaction cell technology (CCT) of inductively coupled plasma mass spectrometry (ICP-MS) International Journal of Coal Geology vol. 124 pp. 1–4.
[5] Altundag H and Tuzen M 2011 Comparison of dry, wet and microwave digestion methods for the multi-element determination in some dried fruit samples by ICP-OES Food and Chemical Toxicology vol. 49 pp. 2800-07.
[6] Enamorado-Baez S M, Abril J M and Gomez-Guzman J M 2013 Determination of 25 trace element Concentrations in biological reference materials by ICP-MS following different microwave-assisted acid digestion methods based on scaling masses of digested samples ISRN Analytical Chemistry vol. 2013 Article ID 851713 14 pages.
[7] Wang H L, Sun D, Wang L, Zhao X and Sun N M 2017 Basic knowledge and application prospect of microwave digestion system Shandong Chemical Industry 46(22) pp. 108+110.
[8] Liu S, Li X 2013 Study on calibration method for microwave digestion system Electronic Product Reliability and Environmental Testing 2013 vol. (9) pp. 223-226.
[9] Li G 2017 Research on calibration of microwave digestion equipment Metrology & Measurement Technique 2017 vol. (9) pp. 80-81.
[10] GB/T 26814-2011 2011 Microwave digestion equipment.
[11] GB/T 18800-2008 2008 Household microwave oven – Methods for measuring performance.