Development of Temperature Calibration Device for Microwave Digestion System in Biochemical Laboratory

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Abstract. As a sample pretreatment device, microwave digestion instrument is widely used in biochemical laboratories. The temperature parameters of the microwave digestion instrument directly decide the accuracy of the experimental results. At present, there is no relevant calibration specification, method or device for microwave digestion system in China. Due to the special working principle of the microwave digestion instrument, the calibration device must be used in the high temperature microwave environment, and the ordinary temperature measuring device will cause ignition, explosion and other phenomena, which will endanger the life and health of the calibration personnel. This article presents a set of calibration device for microwave digestion system, with measuring range of (0~200) ℃, accuracy of no less than ± 0.1 ℃, and resolution of 0.01 ℃. The presented device also solves the potential problems of ignition and explosion, which commonly exist in ordinary temperature measuring devices.

1. Introduction

Microwave digestion technology uses the penetration and activation of microwave to heat the reagents and samples in the closed container, which can increase the pressure in the sample preparation container and the reaction temperature, thus increasing the reaction rate and shortening the sample preparation time. Microwave digestion system can control the reaction conditions to make the sample preparation more accurate, reduce environmental pollution and improve the working environment of laboratory personnel, therefore it has been widely used in biochemical laboratory.[1-8]

At present, microwave digestion systems can mainly be divided into two categories: non-closed microwave digestion system and closed microwave digestion system. At present, closed microwave digestion system has occupied a more than 80% market share, for it can improve the reaction efficiency by significantly increasing the reaction speed, and provide certain flexibility in operation. Generally, the closed pressurized microwave digestion system contains 6~10 closed digestion tanks. Adding reagent to a high temperature and closed container for digestion not only greatly reduces the sample digestion time, but also achieves the advantages that traditional sample processing methods can not match, such as the lowest reagent dosage and background value with a complete recovery rate.

Temperature is the key parameter for the success of the microwave digestion reaction. If the temperature and the pressure in the tank is too high, it will result in volatilization of effective components which may cause tank explosion endangering the safety of users[9]; if the temperature and the pressure in the tank is too low, it will lead to incomplete digestion which will affect the subsequent experimental results. Therefore, regular calibration of the temperature field of microwave digestion system can not only ensure that the consumables are used in a relatively stable state and extend the service life of the system and consumables, but also guarantee the accuracy of the experimental results.

However, there is no calibration specification for microwave digestion system in China at present, and the relevant national standard GB/T 26817-2011 *Microwave Digestion Equipment* only specifies maximum permissible temperature indication error, but without an effective calibration method. Therefore, it is urgent to develop a set of temperature calibration device that can monitor the temperature control of microwave digestion system, realize the wireless real-time measurement of temperature field inside the closed digestion tank, which can ensure the reliability of microwave digestion system in daily use.

2. Design of temperature calibration device for microwave digestion system

The designed temperature calibration device is composed of 5 wireless temperature measurement units. Each unit is consist of temperature acquisition module, power module,

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and microwave shielding module. The structure of the unit is demonstrated in figure 1.

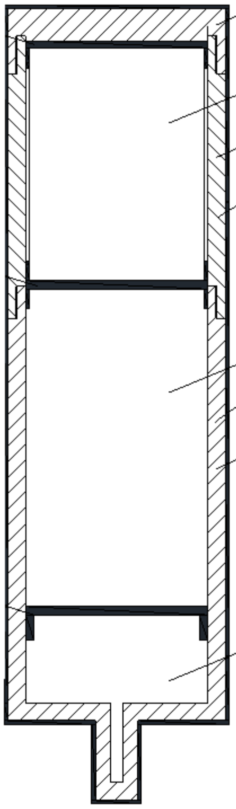

Figure 1. Structure of wireless temperature measurement unit.

2.1 Temperature acquisition module

The temperature acquisition module adopts PT1000 platinum resistance as the high-precision temperature measuring probe, whose accuracy can reach ± 0.01 ℃, and measurement range can meet the calibration requirement of (0~200)℃, with high response speed to the temperature change inside the digestion tank of the microwave digestion system. The temperature acquisition module undertakes the function of temperature measurement, recording and transmission.

2.2 Power module

The power module adopts a high-temperature lithium battery with a stable working range of (0~200)℃. The power module is reinforced with Dow Corning 3104RTV coating in the sealed cavity in order to ensure its stable work status under high pressure of no more than 2MPa inside the closed tank of the microwave digestion system.

2.3 Microwave shielding module

Microwave digestion is conducted in high temperature, high pressure and chemical corrosive solutions, and ordinary metals are prone to melting, deformation and corrosion in such environment. Therefore, the bottom tube, middle tube and upper cover of the wireless temperature measuring unit is made of 304 stainless steel with specific roughness and curvature; and the insulating base plate, insulating middle plate and insulating upper plate is made of PEEK which can completely reflect microwave. The combination of the two materials can protect the PT1000 platinum resistance from high pressure and chemical corrosive solutions. The inner diameter and outer diameter of the bottom and middle tubes are (15.3±0.1)mm and (18.3±0.1)mm respectively, the outer diameter of the upper cover is (18.3±0.1)mm, and the thickness of the top plate of the upper cover is (2±0.1)mm, which can effectively prevent the generation of electric sparks in the closed digestion tank.

3. Calibration method

Start the temperature calibration device, set the sampling time interval not more than 15s, and each unit shall record no less than 36 measurement values respectively. Put the 5 wireless temperature measurement units into a microwave digestion tank as shown in figure 2, add purified water to the digestion tank until the probes of the calibration device are immersed under the water level, seal the digestion tank, and place it on the rotary table. Set the temperature of the microwave digestion system at 50 ℃, 100 ℃ and 180 ℃ respectively, set the temperature rise time for 5min and the constant temperature time for 10min at each temperature point, and start recording the data 1min after reaching the set temperature. 3 temperature parameters (temperature indication error, temperature uniformity, temperature fluctuation) should be calibrated to evaluate the temperature control performance of microwave digestion system.

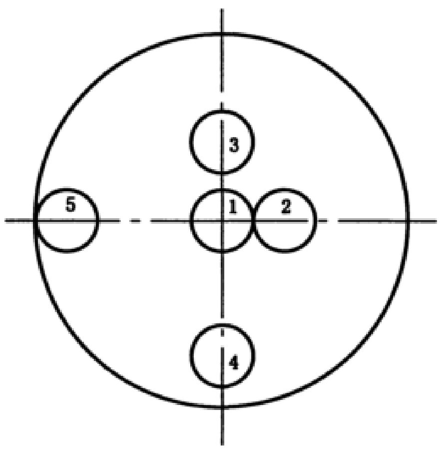

Figure 2. Layout diagram of 5 wireless temperature measurement units.

3.1 Temperature indication error

Calculate temperature indication error Δt_d according to equation (1)[10].

$$\Delta t_d = t_s - \bar{t} \tag{1}$$

Where t_s is the set temperature of the microwave digestion system, and $\bar{t}$ is the average of all recorded data at each temperature calibration point.

3.2 Temperature uniformity

Calculate temperature uniformity Δt_u according to equation (2)[11].

$$\Delta t_u = \frac{\sum_{i=1}^{n}(t_{i\max} - t_{i\min})}{n} \tag{2}$$

Where $t_{i\max}$ is the maximum of all recorded data of one temperature calibration unit at each temperature calibration point, $t_{i\min}$ is the minimum of all recorded data of one temperature calibration unit at each temperature calibration point, and n is the number of measurements of one temperature calibration unit at each temperature calibration point.

3.3 Temperature fluctuation

Calculate temperature fluctuation Δt_f according to equation (3)[11].

$$\Delta t_f = \pm \frac{(t_{f\max} - t_{f\min})}{2}$$

Where $t_{f\min}$ is the maximum of all recorded data of temperature calibration unit 1(as demonstrated in figure 2) at each temperature calibration point, and $t_{f\min}$ is the minimum of all recorded data of temperature calibration unit 1(as demonstrated in figure 2) at each temperature calibration point.

4. Experimental verification of calibration method

Select 11 typical types of microwave digestion systems as the calibration subjects for verification experiment. The calibration results of 11 typical types of microwave digestion systems are demonstrated in table 1.

Table 1. Calibration results of 11 typical types of microwave digestion system.

Type	Temperature(℃)	Parameter	Result(℃)
Multiwave PRO 41HVT56	50	Temperature indication error	0.2
		Temperature uniformity	1.0
		Temperature fluctuation	0.8
	100	Temperature indication error	0.3
		Temperature uniformity	1.0
		Temperature fluctuation	0.8
	180	Temperature indication error	1.2
		Temperature uniformity	2.9
		Temperature fluctuation	0.9
Multiwave GO	50	Temperature indication error	0.5
		Temperature uniformity	0.9
		Temperature fluctuation	0.9
	100	Temperature indication error	0.4
		Temperature uniformity	1.2
		Temperature fluctuation	1.0
	180	Temperature indication error	1.6
		Temperature uniformity	3.1
		Temperature fluctuation	0.7
TOPEX+	50	Temperature indication error	1.3
		Temperature uniformity	1.5
		Temperature fluctuation	0.7
	100	Temperature indication error	2.3
		Temperature uniformity	2.3
		Temperature fluctuation	0.9
	180	Temperature indication error	3.2
		Temperature uniformity	4.2

		Temperature fluctuation	0.6
		Temperature indication error	-1.6
	50	Temperature uniformity	1.1
		Temperature fluctuation	0.9
		Temperature indication error	2.6
CASH COW	100	Temperature uniformity	2.4
		Temperature fluctuation	0.7
		Temperature indication error	3.3
	180	Temperature uniformity	3.9
		Temperature fluctuation	-0.9
		Temperature indication error	1.2
	50	Temperature uniformity	-1.2
		Temperature fluctuation	0.8
		Temperature indication error	1.9
WX-8000	100	Temperature uniformity	1.9
		Temperature fluctuation	0.9
		Temperature indication error	2.9
	180	Temperature uniformity	-4.5
		Temperature fluctuation	-0.5
		Temperature indication error	-0.9
	50	Temperature uniformity	-0.9
		Temperature fluctuation	-0.7
		Temperature indication error	-1.8
WX-7000HP	100	Temperature uniformity	-2.0
		Temperature fluctuation	-0.7
		Temperature indication error	4.3
	180	Temperature uniformity	4.3
		Temperature fluctuation	0.8
		Temperature indication error	-0.5
	50	Temperature uniformity	-1.4
		Temperature fluctuation	-0.8
		Temperature indication error	-2.5
MARS 6	100	Temperature uniformity	-2.2
		Temperature fluctuation	-0.6
		Temperature indication error	-3.0
	180	Temperature uniformity	-4.0
		Temperature fluctuation	-0.8
		Temperature indication error	1.4
	50	Temperature uniformity	1.1
		Temperature fluctuation	1.0
		Temperature indication error	-1.5
MARS 5	100	Temperature uniformity	2.0
		Temperature fluctuation	1.0
		Temperature indication error	-4.0
	180	Temperature uniformity	-4.4
		Temperature fluctuation	-0.9
		Temperature indication error	1.2
ETHOS ONE	50	Temperature uniformity	-1.2
		Temperature fluctuation	0.9
	100	Temperature indication error	2.3

		Temperature uniformity	2.3
		Temperature fluctuation	0.9
		Temperature indication error	4.1
	180	Temperature uniformity	4.1
		Temperature fluctuation	0.9
		Temperature indication error	0.8
	50	Temperature uniformity	-1.3
		Temperature fluctuation	-0.8
		Temperature indication error	1.8
Milestone ETHOS	100	Temperature uniformity	1.9
		Temperature fluctuation	0.7
		Temperature indication error	3.8
	180	Temperature uniformity	3.3
		Temperature fluctuation	0.7
		Temperature indication error	-0.5
	50	Temperature uniformity	0.8
		Temperature fluctuation	0.9
		Temperature indication error	-1.1
ETHOS UP	100	Temperature uniformity	-1.7
		Temperature fluctuation	0.9
		Temperature indication error	-3.6
	180	Temperature uniformity	-3.8
		Temperature fluctuation	-0.8

As we can see from table 1: the temperature indication error calibration results of 50℃ can meet the requirement of GB/T 26817-2011 *Microwave Digestion Equipment* of no more than ±2℃, the temperature indication error calibration results of 100℃ can meet the requirement of GB/T 26817-2011 *Microwave Digestion Equipment* of no more than ±3℃, and the temperature indication error calibration results of 180℃ can meet the requirement of GB/T 26817-2011 *Microwave Digestion Equipment* of no more than ±5℃; both temperature uniformity and fluctuation calibration results can meet the enterprise standards of manufacturers. The feasibility and applicability of the calibration device and method have been proven.

5. Conclusion

This article designs the temperature calibration method of microwave digestion system on basis of research on the inner temperature field of the system, and presents a wireless temperature calibration device. In order to ensure the stable working performance of the calibration device in high temperature and high pressure environment, 304 stainless steel and PEEK coating are adopted in the construction of the device. The temperature maximum permissible error of the device is less than 0.1 ℃. The calibration results show that the device meets the temperature calibration requirements of microwave digestion system. The follow-up study will further optimize the design of the device, reduce the size of the temperature measurement unit, improve the portability of the device, and upgrade the calibration scheme and accuracy.

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