

## STANDARD OPERATING PROCEDURE (SOP) FOR MICROWAVE DIGESTION SYSTEM (MDS-10)

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**ABSTRACT:** Microwave digestion system plays very important role for digestion on different samples such as tissues, blood, hair, nails, rocks, minerals etc during sample preparation for analytical purposes. Its use is readily understood from breadth of its applications. The range of material that can be analyzed by Microwave digestion system is essentially unlimited. Applications are found in diverse fields such as forensic science, pharmaceuticals, food and agriculture, biological and clinical chemistry, environmental chemistry and many others. Standard operating procedure for Sineo make&model MDS10 Microwave digestion system have been developed. The different steps involved for smooth running of microwave digestion system has been successfully explained.

**Key Words:** Standard operating procedure(SOP), microwave digestion, MDS-10, blood, tissue, urine, etc.

### INTRODUCTION:

For analysis of different elements in any material such as solid, liquid etc, the first step is to bring the element in solution form which is achieved by acid digestion procedure. The goal of every digestion process is therefore the complete digestion of the solid by avoiding loss or contamination of the analyte.<sup>1</sup> Digestion is carried out by open acid digestion or closed digestion. There are numerous advantages of the closed procedure over open digestions such as low acid consumption, less time, good digestion quality, no loss of volatile element such as Hg, Pb etc. and no contamination risk.<sup>2</sup> All electromagnetic waves are sky waves which propagate at the speed of light and transport energy without loss in a vacuum and

with loss in dielectric material. They show the same effects as light such as interference, diffraction, refraction, reflection, polarizability. Microwaves are high-frequency electromagnetic waves in the same frequency band as radar waves. Only four microwave frequencies are permitted for industrial and scientific use. Out of these, 2.45 GHz is the most frequently used.<sup>3</sup> Microwave digestion is widely used technique for the digestion of samples for metal analysis using GFAA, ICP-OES and ICP-MS etc. Since it operates at far higher temperature and pressure than the open digestion, it can be applied to a much wider range of samples, producing a clear digested sample from even the most refractory sample type.<sup>4</sup> Naturally the employed vessels must be resistant to the acids and reagents being used.<sup>4</sup>

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Glass except for solution containing HF, PP, PE are all suitable for vessel in which digestion solutions are to be stored. Commonly used reagents are nitric acid (HNO<sub>3</sub>), hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), hydrochloric acid (HCl), nitrohydrochloric acid, hydrofluoric acid (HF), sulphuric acid (H<sub>2</sub>SO<sub>4</sub>). Acid digestion whether open or closed pressure digestion are carried out with the aid of a wide variety of reagents. Aside from the various mineral acids, other reagents such as hydrogen peroxide, potassium peroxide sulphate, boric acid, and many more are also used. The selection of the reagents or the preparation of a reagent mixture depends on the sample to be digested. Organic sample materials are generally decomposed into carbon dioxide with the help of oxidizing acid such as nitric acid and hydrogen peroxide. <sup>5</sup>

#### **PURPOSE**

To develop the standard operating procedure for Sineo MDS-10. To ensure its compliance with the provision of Good Laboratory Practice regulations.

#### **SCOPE**

Describes the finest details of the steps to be followed in one of the simplest and precise way required in the analytical technique of Sineo MDS-10

#### **RESPONSIBILITIES**

All the scientific staff members carrying out the Sineo MDS-10 are responsible for strictly adhering to the procedures given in this text.

#### **GENERAL OPERATIONAL CONDITION OF MDS-10<sup>6</sup>**

1. Operating temperature is 5 to 350°C
2. Relative humidity is less than 60%
3. Power plug must be properly grounded.

4. High strength frame reaction vessel sustained pressure 15MPa & sustained temperature of over 300°C.

5. The sample dissolving cup general assembly is made from imported high strength TFM and able to resist the corrosion of all acids and alkalis, with the highest working temperature of 260°C.

6. Max. sustained pressure is 5MPa (700psi).

7. Temperature control range from room temperature to 260°C.

#### **ASSEMBLING OF DIGESTION VESSELS<sup>6</sup>**

1. Sample dissolving cup must be washed from ultrapure/double distilled water prior use.
2. Add sample and solvent to sample dissolving cup.
3. Press the expander of sealed cover down to the end through the sealed cover to expand the edge film of the cover.
4. Press "TURN" on the panel to make the desired position to fix the vessels.
5. When inserting Master vessel temperature and pressure the sensors align RED POINT positions.

#### **DISASSEMBLING OF DIGESTION VESSELS<sup>6</sup>**

1. Once the program gets finished, cooling start automatically, wait for temperature to drop to 45°C.
2. Take out the master vessel and transfer it into ventilation cabinet. Pull out the temperature sensor probe immediately and insert the temperature measuring seal tube setting rod.
3. Punch the sample dissolving cups by punch plate to take out sample dissolving cup from digestion frame.

#### **CLEANING OF DIGESTION VESSELS<sup>6</sup>**

1. Soak the sample dissolving cups and sealed covers in clean running water or diluted acid. Do not soak the sealed cover of temperature

measuring vessel at same time. No water shall enter into the tube, otherwise the temperature sensor will be damaged.

2. Wipe the vessel frames and safety bolts by soap water after each use to extend the life time.

### OPERATION INSTRUCTION OF MAIN UNIT<sup>6</sup>

1. The warming-up period of the device is 10 min through temperature ramping, which was included in the program.

2. In the “Method selection” interface, press “Enter” once to switch over to “method directory”.

3. After enter put method number at Method Selection, press enter key again.

4. Setup page. Press “Set” key, and “1” will be displayed under “N” (Step 1), then put value of temperature (T°), time (t in min) and power (W) and create sequence

Create and save programme.

5. Numbers from 04~16 represent power values from 400W to 1,600W.

6. During operation, “TIME” is counting down. “PRES” reflects real pressure and “TEMP” reflects real temperature. The “Page” key can be pressed at any moment to switch to pressure rise or temperature rise curve page.

7. Select instrument power according to table1.

**Table1: Vessel quantity and instrument power<sup>6</sup>**

Weight of sample	Total quantity of sample	Quantity of Vessel	Microwave power
0.1-1.0	5-20 ml	1-2	400
0.1-1.0	5-20 ml	3-4	400-600
0.1-1.0	5-20 ml	6-8	600-800
0.1-1.0	5-20 ml	8-10	800-1000
0.1-1.0	5-20 ml	15-20	1000-1200

### CREATING NEW PROGRAM

1. Switch on the instrument from the button provided in the back of the instrument.

2. Choose the program no (00 to 54) and press enter key( If method is already established then just run the machine using the green button otherwise press the reset button and enter the new values according to the new method).

3. Enter the required parameter (i.e. temp, time and power) for example – for power 400 w press 04, for 600 watt press 06.

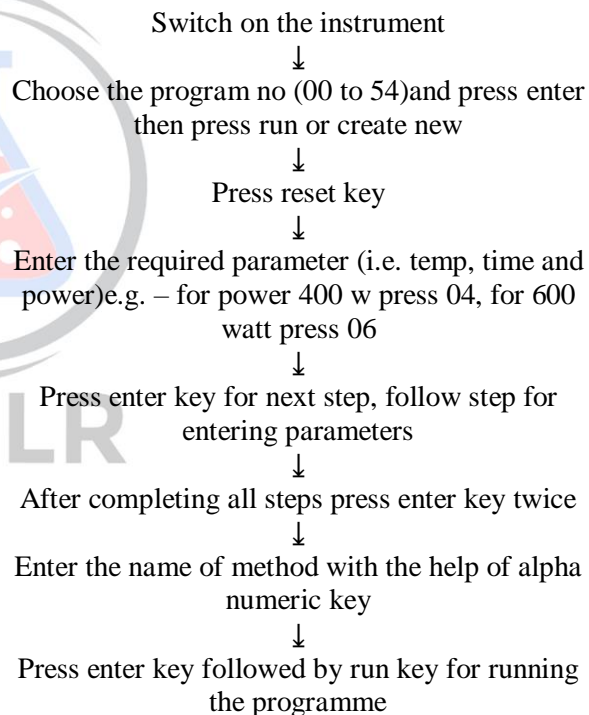
4. Press enter key for next step and follow step for entering parameters.

5. After completing all steps press enter key twice.

6. Enter the name of method with the help of alpha numeric key.

7. Press run key for running the program.

### Flow chart 1: Creating new programme



### STANDARD OPERATING PROCEDURE OF THE PROGRAMME

1. Switch ON MDS 10, wait for the method window to appear.

2. Instrument will stop at Method selection.

3. From method selection menu, enter programme number that is to be run or create new.
4. To run the already created (old programme) program, press run key after loading the program.
5. To create or edit programme press 'reset' key enter (T) temperature as per the sample type given in application manual, (t) time in minute & (W) microwave power then press enter to go in next line again enter the values according to the type of sample and quantity of vessels then press enter twice to write program no. and name. Press enter to save the program
6. Prepare sample (as per application), use expander to expand the mouth of vessel as a precaution.
7. Place sample vessel inside instrument cavity on turntable and close the door.
8. Select desired programme.
9. Press green key to run the programme.
10. After digestion, cooling will start automatically and wait till temperature reaches upto 45°C.
11. Open the door and take out the vessels.
12. Open vessel in fume hood after vessel attains room temp (use punch plate).
13. Remove temp sensor from master vessel and insert setting rod inside it.
14. Make up sample as required.

After digestion cooling start automatically and wait till temp reaches upto 45°C  
 ↓  
 Open the door  
 ↓  
 Take out sample vessel  
 ↓  
 Remove temp sensor from master vessel and insert setting rod inside it  
 ↓  
 Open vessel in fume hood after vessel attain room temp (use punch plate)  
 ↓  
 Make up sample as per requirement

## APPLICATION OF MICROWAVE DIGESTION SYSTEM

### 1. Digestion of Blood

Digestion reagent: HNO<sub>3</sub> 69%, H<sub>2</sub>O<sub>2</sub> 30%  
 Sampling quantity: 1.0 ml  
 Digestion mixture: 1 mL blood, + 4ml HNO<sub>3</sub> + 1 mL of H<sub>2</sub>O<sub>2</sub>.  
 Digestion programme : Table 2

**Table 2: Programme for digestion of Blood**

Step	Temp	Time	Power of 1-2vessel	Power of multi-vessel Given in Table No 1
	°C	°C	Watt	
1	120	10	400	
2	150	10	400	
3	180	10	400	
4	200	10	400	

### Flow chart 2: Standard Operating Procedure of MDS 10

Switch on instrument  
 ↓  
 Prepare sample (as per application) use expander  
 ↓  
 Place sample vessel inside instrument cavity on turntable  
 ↓  
 Select desired programme  
 ↓  
 Press green key to run the programme  
 ↓

### 2. Digestion of Tissue

Digestion reagent: HNO<sub>3</sub> 69%, H<sub>2</sub>O<sub>2</sub> 30%  
 Sampling quantity: 0.5 g  
 Digestion mixture: 0.5 g Tissue + 4 ml HNO<sub>3</sub> + 1 mL of H<sub>2</sub>O<sub>2</sub>.  
 Digestion programme : Table 3

**Table 3: Programme for digestion of Tissue**

Step	Temp	Time	Power of 1-2vessel	Power of multi-vessel Given in Table No – 1
	°C	°C	Watt	
1	120	10	400	
2	150	10	400	
3	180	10	400	
4	200	15	400	

### 3. Digestion of Hair

Digestion reagent: HNO<sub>3</sub> 69%  
 Sampling quantity: 0.1g  
 Digestion mixture: 0.1 g Hair + 4 ml HNO<sub>3</sub>.  
 Digestion programme : Table 4

**Table 4: Programme for digestion of Hair**

Step	Temp	Time	Power of 1-2vessel	Power of multi-vessel Given in Table No 1
	°C	°C	Watt	
1	130	10	400	
2	150	10	400	
3	180	15	400	
4	200	15	400	

### 4. Digestion of Nail

Digestion reagent: HNO<sub>3</sub> 69%  
 Sampling quantity: 0.1 g  
 Digestion mixture: 0.1 g Nail + 4 ml HNO<sub>3</sub>  
 Digestion programme : Table 5

**Table 5: Programme for digestion of Nail**

Step	Temp	Time	Power of 1-2vessel	Power of multi-vessel Given in Table No 1
	°C	°C	Watt	
1	130	10	400	
2	150	10	400	
3	180	10	400	
4	200	15	400	

### 5. Digestion of Urine

Digestion reagent: HNO<sub>3</sub> 69%  
 Sampling quantity: 5.0 ml  
 Digestion mixture: 5 mL Urine + 1.5 ml HNO<sub>3</sub>  
 Digestion programme : Table 6

**Table 6: Programme for digestion of Urine**

Step	Temp	Time	Power of 1-2vessel	Power of multi-vessel Given in Table No – 1
	°C	°C	Watt	
1	130	10	400	
2	150	10	400	
3	180	5	400	
4	200	5	400	

### DO AND DON'T FOR MICROWAVE DIGESTION SYSTEM<sup>6</sup>

#### Do's

1. Must expand the sealed cover before each operation to ensure the excellent sealing.
2. Must keep the sealed cover and the inner wall of sample dissolving cup clean.
3. The sealed cover edge is very gentle. Handle with care to avoid any damages.
4. While inserting or pulling out a plug, support the junction box by other hand.
5. Temperature probe must be pulled out at about 45°C and 01 MPa pressure. Insert the setting rod immediately after temperature probe taken out.
6. it's recommended to set the working temperature within 240°C.
7. The operator shall not leave immediately once heating starts but carefully observe the temperature rise in the first working step, especially the temperature change from 110 to 130°C. If the temperature rises regularly in this interval, it will be judged that the master vessel is well sealed and working normally.
8. Any emergencies in between digestion press red key to stop.
9. Sample vessel must be opened in fume hood.
10. Wash the vessel frames and safety bolts by soap solution followed by water after each and every use.

11. Machine should be run with voltage stabilizer only.
12. Clean each vessel from ultrapure/double distilled water after every use to avoid contamination.
13. Always put the reference material in the temperature sensor vessel to avoid contamination.
14. Always keep the vessels on the turntable after balancing the weights from opposite sides.
15. Always use the Spencer to open the vessels as it releases the pressure and it also avoids burning of hands from fumes of acid.

### Don'ts

1. Don't screw the safety bolt too much onto the sealed cover top this may cause the vessel leakage and abnormal burst of the explosive bolt.
2. Never use the spanner with full strength. It may destroy the safety bolts and spanner.
3. During run don't open the door.
4. No water shall enter into the temperature sensor tube otherwise the temperature sensor tube will be damaged.
5. Don't run the turntable manually. Always use the turntable rotator button to move it.

### TROUBLE SHOOTINGS

1. If cap is not finely fitted then use expander.
2. If vessel is not properly cleaned then use diluted acid followed by ultrapure water/distilled water.
3. If vessel explodes during run, immediately close the machine using the button behind the machine and open it after 1-2 hours when temperature slows down to room temperature.
4. Always set the power in the method according to quantity of vessels mentioned in table 1.

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