MILESTONE
HELPING CHEMISTS

DRN-41

Operator Manual

MA166-001

MILESTONE DRN-41 HIGH THROUGHPUT ROTOR USER MANUAL

Thank you for choosing our high throughput rotor for the microwave digestion system.

We are sure that you will be completely satisfied with the performance of this new accessory entering your laboratory.

The valid version of this manual is English.
In case of translation in other languages, please refer to the original version.

We invite you to read carefully this user manual and to keep it in reach for convenient and fast consulting.
For any possible clarification or any request for assistance please contact either Milestone Srl at the following address:

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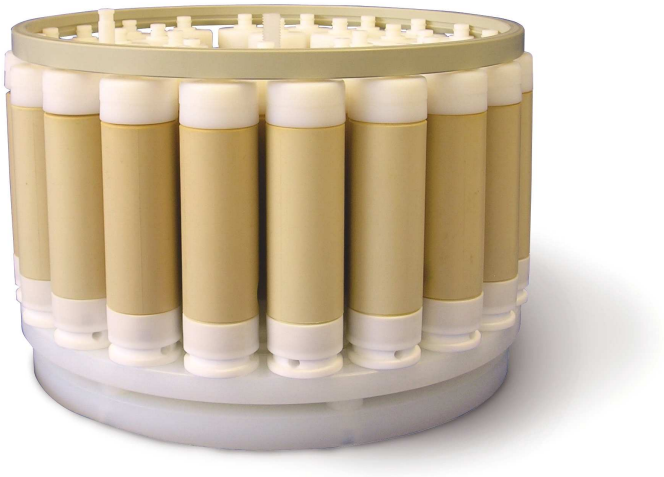
DRN-41 HIGH THROUGHPUT ROTOR

The DRN-41 rotor contains 41 pressure reactors for digestion.

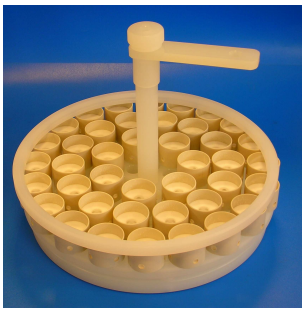


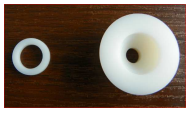
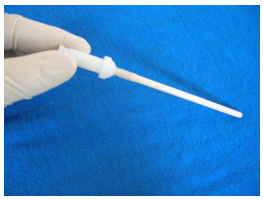
DRN-41 TECHNICAL SPECIFICATION

Sample capacity	41
Test temperature	300°C
Working temperature	**200°C**
Test pressure	100 bar
Working pressure	**50 bar**

DRN-41 ROTOR FOR TEMPERATURE CONTROL

The DRN-41 rotor is composed by the following items:

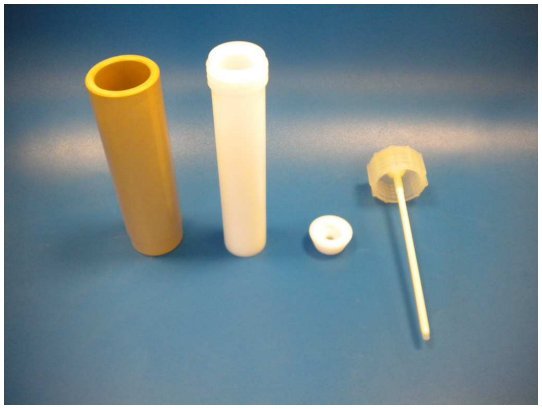
Qty	Picture	P/N	Description
1		MUP04100	Polypropylene rotor body with cover
41		HB00055FC2	70 ml TFM vessels
41		DD00107	PFA cover with hole
41		DD00104FC2	TFM cap
41		DD00073	Safety shield
41		OR0018	O-ring

Reference vessel			
Qty	Picture	P/N	Description
41		HB00055FC2	70 ml TFM vessels
41		DD00107	PFA cover with hole
41		DD00104FC2R	TFM cap
41		DD00073	Safety shield
1		MUR00011/M6	Thermowell for vessel 70 ml

Following pictures shown a standard complete vessel and the complete reference vessel of the DRN-41 rotor:

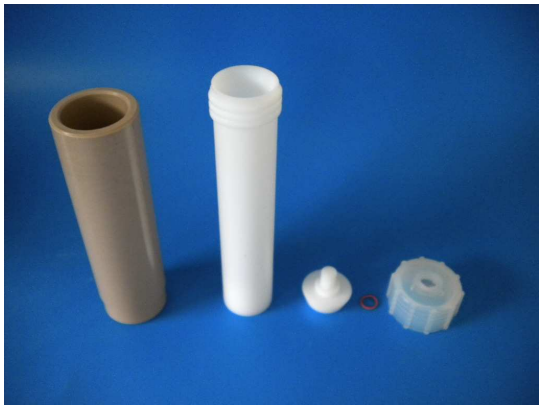
N.1 Reference pressure reactor

Safety shield
PFA vessel of 70 ml
TFM cap
PFA cover
Safety valve with thermowell

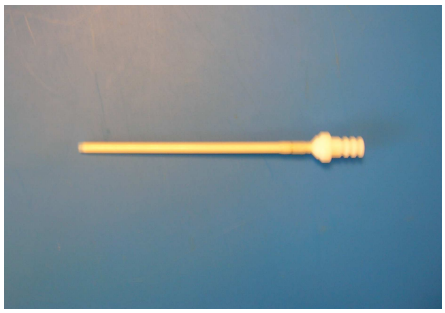


N.40 Standard pressure reactor

Safety shield
PFA vessel of 70 ml
TFM cap
PFA cover
Safety valve



Thermowell with thread (only for 050214/M6 ATC-400 sensor):



HOW TO OPERATE WITH THE DRN-41 rotor

SAMPLE WEIGHING, REAGENTS ADDITION AND PRESSURE REACTORS ACCOMODATION

Note: before starting with this procedure, all the parts of this rotor must be cleaned, dried and checked. **Always wear protection glasses, coat and gloves when operating with the Microwave system.**

Place a TFM vessel on the balance plate.

Tare the vessel and directly weigh the sample by placing it inside the vessel.
Try to place all the samples on the bottom of the vessel.

Add suitable reagents to the sample. When part of the sample is left on inner wall of the vessel, try to wet it by adding acids drop by drop.
Gently swirl the solution, to homogenize the sample with the reagents.
In case of a reaction appear, leave the vessel open under fume hood.

WARNING: The minimum volume is 10 ml in each reactor.

Place the TFM cover on the vessel

Tight by hand the Safety valve until completely closed.

Repeat the same procedure for all reactors.

Introduce the pressure reactor into the safety shield.

SETUP OF THE REFERENCE PRESSURE REACTOR, USED FOR TEMPERATURE CONTROL

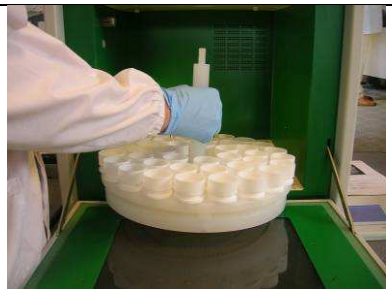
Notice the reference cover, provided with one port, and the thermowell.
The thermowell is made of ceramic, and it is Teflon coated to avoid any sort of corrosion.
Place the Teflon cover on the vessel; push firmly to the end and check that the cover fits well onto the vessel.

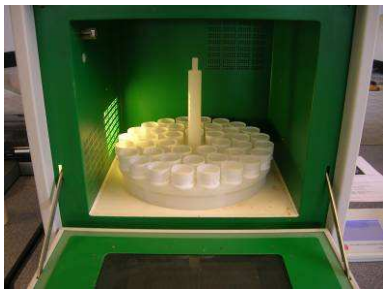
WARNING: the reference vessel must contains the same sample (type and amount) of the standard vessels

Introduce the ceramic thermowell (p/n MUR0011/M6) as shown below and tighten by hand the Safety valve until completely closed.

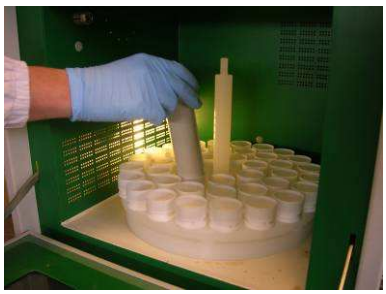
ROTOR AND VESSELS ACCOMODATION AND SENSOR PLUG IN

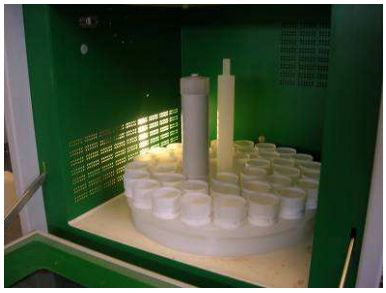
Place the rotor inside the microwave cavity.





Introduce a standard vessel into the rotor body

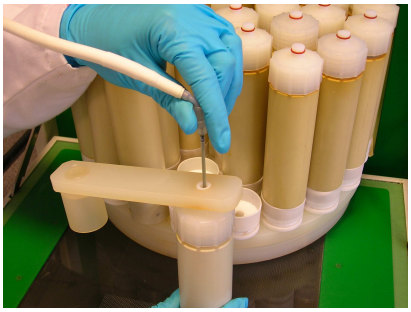




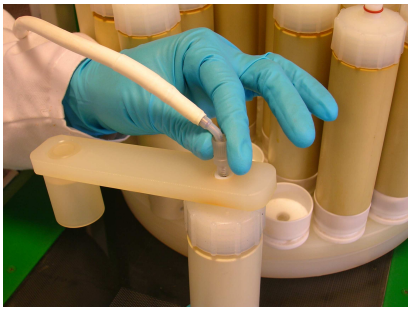
Introduce all the standard vessels into the rotor.

Place the reference holder on the reference vessel
The reference holder is used to fix the reference vessel in order to prevent its fall, therefore must be always used.

Introduce the ATC-400 sensor (050214/M6) into the thermowell (MUR00011/M6)



…and tight the PTFE adapter of the sensor into the thermowell



WARNING :

Always introduce the ATC-400sensor in the middle ring, even if TEMP-SURE is installed

NOTE
The temperature control will be done by the ATC-400. If TEMP-SURE is installed it will display only the 20 external temperatures (infrared temperatures) of the external ring.

Push the reference holder into the column

Tight the thread ring onto the central column

Place the ring cover on the rotor. The sensor must pass trough the ring.

Plug in the ATC-400 Temperature sensor to the device control of the Microwave unit.

WARNING: Touch only the metal connector. As shown in the previous picture, it is important to handle the sensor only through the connector. This will avoid any possible damage of the sensor.

Your DRN-41 rotor is now ready to operate!

COOLING DOWN AND UNCAPPING PRESSURE REACTOR

After an heating cycle has been completed, allow a 20 minutes cooling time (for full rotor) of the pressure reactors into the microwave cavity.

WARNING: Due to a substantial expansion of the plastics used at high temperature, the removal of the ATC sensor from reference pressure reactor should be done only when the inner temperature of the vessel is approximately 180°C.

Once the program is completed, high temperatures are achieved inside the pressure reactors, according with the digestion procedure applied.
It is therefore necessary to cool down the pressure reactors before removing from the rotor body. The rotor may be air cooled inside the Microwave unit and in such case the cooling time is around 30 minutes (for full rotor)

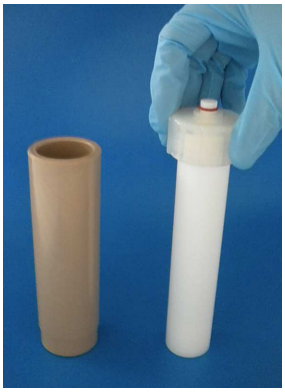
WARNING: For safety reasons, do not remove the DRN-41 rotor if it is too hot.

Place the reactors under a fume hood and open only when the room temperature has been achieved (50-60°C as maximum).

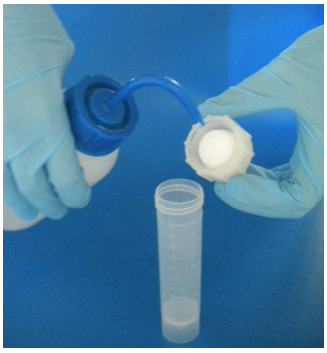
UNCAPPING OF A STANDARD VESSEL

Remove the vessel from the rotor

Remove the TFM vessel from the safety shield



Un-tight and remove the cover and rinse it using distilled water



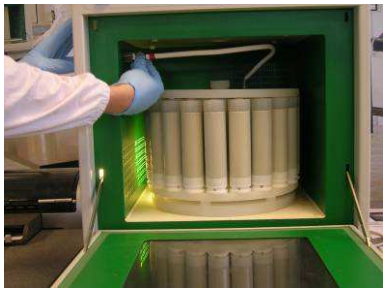
Rinse the vessels with distilled water

Transfer the digested solution into a suitable volumetric flask

Repeat the operation for all standard reactors of the rotor.

UNCAPPING OF THE REFERENCE VESSEL

Disconnect the temperature sensor

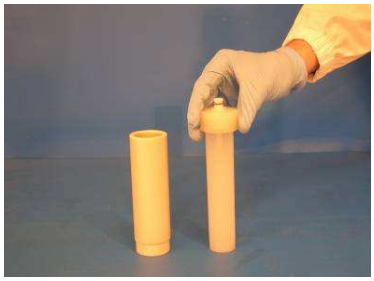


Remove the rotor ring

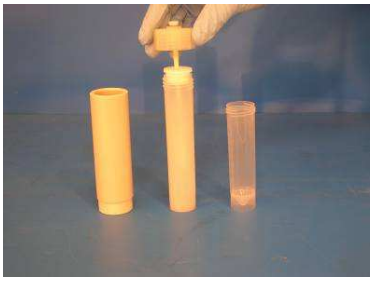
Remove the complete reference vessel and the reference holder from the rotor

Remove the sensor by losing the threaded adapter and the reference holder

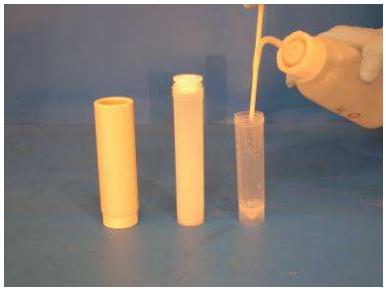
Remove the reference vessel from the shields



Open the vessel



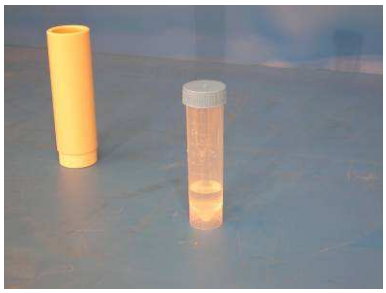
Remove the acids droplets from the termowell with a pipette using distilled water



Rinse the vessels and the TFM cap

Transfer the digested solution into a suitable volumetric flask

Your sample is now ready to be analyzed.



GUIDELINES LINES FOR ACID DIGESTION

Milestone's DRN-41 rotor has been designed for increasing the sample throughput in modern analytical laboratories.

For a proper use of rotor, Milestone strictly recommends to follow these basic rules.

APPLICATION

Milestone DRN-41 rotor can be used for the following samples:

- Soils
- Sands
- Sediments
- Sludge
- Easy organics like vegetables, biological tissues and foods (less then 20% fat content)
- Water, waste water

Since Milestone's DRN-41 rotor has a working pressure of 50 bar, the typical sample size used in a single pressure reactor is 200 mg of dry organic matter. For further information, please contact your local Milestone distributor.

WARNING: For the digestion of water amount higher then 20 mL, it is recommended the use of ASM-400 for the stirring of reaction mixture during the digestion.

REAGENTS

All digestion processes have to be performed using a minimum amount of 10 mL reagents in each vials using the internal temperature control.

For a better heating homogeneity and microwave distribution, it is important that all pressure reactors are filled with the same amount of reagents. Basic acid mixtures for sample digestion, in according to sample characteristics are the following:

- Organic samples
  8 mL HNO3 65% - 2 mL H2O2 30%
- Soils, sands, sediments, sludge
  10 mL HNO3 65% (US EPA 3051)

Anyway, Milestone's application specialists are pleased to support you giving a suitable application notes for your specific sample.
For any question please contact your local Milestone Product Specialist or distributor.

Typical procedure:

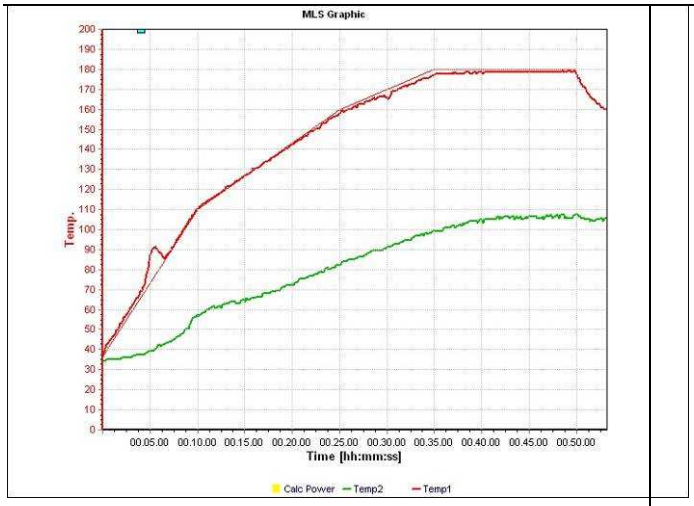
Sample amount : 0.2-0,5 g of soil sample
Reagent : 8 mL HNO3 65% - 2 mL H2O2 30%
Vessel number: 41

Microwave program for EasyCONTROL and EasyWAVE software:

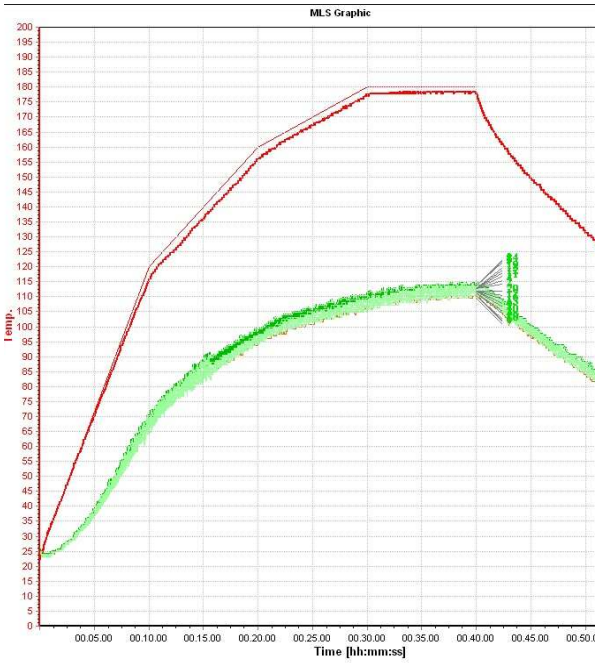
1 step
10 minutes 110°C up to 1000/1200/1500 W*
2 step
15 minutes 160°C up to 1000/1200/1500 W*
3 step
10 minutes 180°C up to 1000/1200/1500 W*
4 step
15 minutes 180°C up to 1000/1200/1500 W*

* Set the maximum power of your unit.

Ventilation
20 minutes



Example of Temperature profile using Milestone TEMP-SURE



Noticed that 20 temperature profiles (green lines) are showed for the 20 vessels of the outer ring. The red line shown the ATC-400, that is the internal temperature control of the system.

Notes

This procedure is only a guideline and it may need to be modified or changed to obtain the required results on your sample.

Suggested microwave program for 10 vessels:
1 step
15 minutes 180°C up to 1000/1200/1500 W*
2 step
15 minutes 180°C up to 1000/1200/1500 W*

Suggested microwave program for 20 vessels:
1 step
10 minutes 120°C up to 1000/1200/1500 W*
2 step
10 minutes 180°C up to 1000/1200/1500 W*
3 step
15 minutes 180°C up to 1000/1200/1500 W*

Suggested microwave program for 30 vessels:
1 step
10 minutes 120°C up to 1000/1200/1500 W*
2 step
15 minutes 180°C up to 1000/1200/1500 W*
3 step
15 minutes 180°C up to 1000/1200/1500 W*

MAINTENANCE OF DRN-41 ROTOR

As general rules:

- Do not use any organic solvent to clean or dry any part of the rotor, with the exception of the TFM Teflon covers and of the TFM vessels.
- Dry thoroughly every component including the vessels with paper towel or cloth, before placing the rotor in the microwave unit.
  Any moisture or reagents trace would heat up in the microwave field and it could possibly damage the components.
  This is especially true if any trace of water or acid/solvent are left between the internal wall of the safety shields and the external walls of the vessels.
- Do not use ice to cool down the rotor after microwave heating, as the thermal shock could damage the safety shield.

WARNING: DO NEVER IMMERSE THE THERMOWELL INTO WATER, ACID OR INTO ANY OTHER SOLVENT.

- To wash all kind of vessels, it is advisable to use the following procedure:

ROTOR CLEANING PROCEDURE

1. Add 4 mL of bi-distilled water and 6 ml of HNO3 65% in each vessel, or 10 ml of HNO3 65%
2. Start the following microwave program (41 pressure reactors): 25 minutes up to 160°C @ 1000 Watt, 10 minutes at 160°C @ 1000 Watt. Increase the power up 1200 or 1500W in according to your unit.
3. Cool down and open the pressure reactor.
4. Rinse the vessels with bi-distilled water.
5. Now the vessels are cleaned and ready for a new digestion

SAFETY SHIELDS

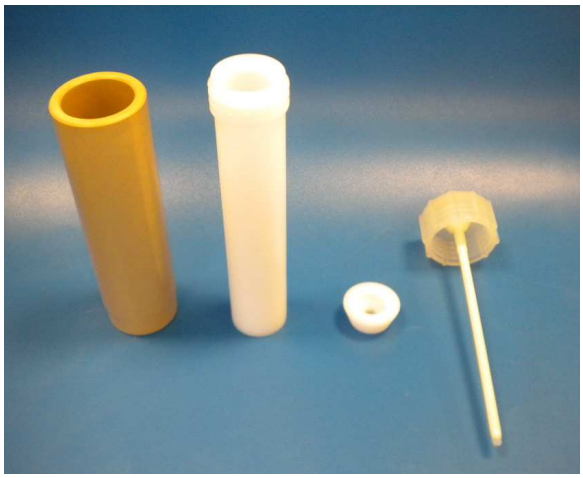
After long use the safety shields may show some swelling due to the high heating conditions.
The safety shield lifetime is estimated to be hundreds of digestions.

It is important check for visible deformation, crack or any other notice of corrosion of the safety shields, replacing them if necessary.

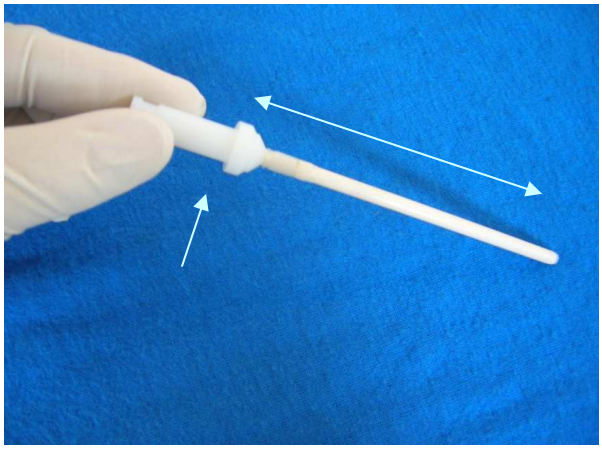
Periodically clean the shields with tap water in order to remove eventual acid drops.
Dry the shields before using them.
Replace the shields if evidence of corrosion is clearly noticed on the walls and on the threads.

REFERENCE PRESSURE REACTOR

The next pictures shows the 70 ml reference pressure reactor complete.



Inspection of the thermowell is very important in order to prevent leakage of reagents and consequently damage of the sensors. When cracks in the ceramic thermowell appear, it must be replaced



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