

CDS Cryo-mill Manual



CDS Analytical
A LabTech Company

Cryo-mill for Polymers

Hardware

A distinct advantage of analytical pyrolysis GC-MS (PY-GC-MS) in polymer analysis is a simplified sample preparation process by skipping the solvent extraction and concentration step. However, the challenging hurdle in the sample preparation for PY-GC-MS is to homogenize the polymer samples, which would determine the reproducibility of the following sample runs. As the rule of thumb for PY-GC-MS, more homogenized samples would yield more reproducible data. Among various homogenizers, cryogenic ball mill has been proven to be a powerful tool in homogenizing thermally sensitive and elastic polymers. CDS Analytical has designed an economical and ready-to-use cryo-mill solution for such applications.

The Cryo-mill is composed of the following hardware as shown in Figure 1

- (1) Cryo-mill
- (2) Liquid nitrogen reservoir
- (3) Metal tongs
- (4) Touch screen pen
- (5) 110V Power adapter

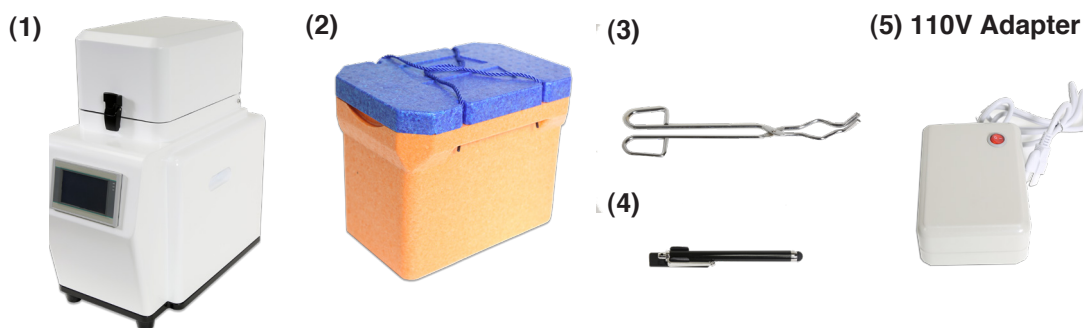


Figure 1: Cryo-mill hardware

A 110V power adapter is provided in the package. If you have a 110V power outlet, the power cord of the cryo-mill needs to be plugged into the power adapter, and then the power adapter needs to be plugged into the power outlet. For 220V power line, plug the cryo-mill directly into the power outlet.

Four 5 ml capacity titanium sample jars are provided on a sample rack (Figure 2 Left). Each jar is composed of a sample grinding cup and a threaded cap, and is engraved with a number from 1 to 4. Five 9.6 mm stainless steel balls (Figure 2 Center) are supplied. The red o-rings (Figure 2 Right) packaged with the stainless steel balls are only for over time (>10 minutes) grinding at room temperature to compensate the shift of the jar cap during grinding, and is not recommended for any cryogenic applications.



Figure 2: Cryo-mill sampling accessories

Principles of Operation

The Cryo-mill adopts the ball milling technique by impact and friction in a sample jar pre-cooled by liquid nitrogen, which (1) lowers the glass transition temperatures of a polymer sample to make the sample more fragile to grind, (2) preserves the volatile additives in a polymer sample from evaporation during the grinding process. Four 5 mL sample jars are mounted on a sample rack that is driven by an electric motor through a metal shaft to introduce vertical oscillation of the grinding ball for crushing, mixing,

Installation

Warning

(1) Unpack carefully. The cryo-mill is as heavy as 60 lbs and requires two people to lift. When unpacking, lift the package box vertically to remove packaging materials, and then move the cryo-mill to your work bench by using two people.



Figure 3: Unpacking Cryo-mill

(2) Tilt the cryo-mill slowly to lay the unit down on a flat work bench in order to expose the bottom. Use the supplied wrench to remove four transportation bolts.

Warning

Sever damages occurs without removing transportation bolts



Figure 4: Remove 4x transportation bolts

(3) Lift the cryo-mill up to stand on the rubber feet. The cryo-mill is designed to work indoors with sufficient ventilation for liquid nitrogen. The working temperature for cryo-mill is 50 °F to 100 °F (10 °C to 40 °C).

(4) For 110V cryo-mill, plug the power cable into the power adapter supplied.

(5) Plug the power cable into the power outlet.

Conditioning Sample Jar

The sample jar supplied is made of titanium which has superior tensile strength to minimize metal debris from grinding process. Before using the cryo-mill for real samples, it is required to complete a conditioning process that further hardens the inner surface of the sample jar.

(1) Wash the sample jar, cap and grinding ball throughly by ultrasonic wash

(2) Load 50 to 60 mg of Polyethylene pellet in the sample jar with the grinding ball and go through the 8-cycle default grinding program. This step could be done at ambient temperature.

(3) The ground powder will be dark and it is normal

(4) Repeat step 1 to 3 again till the color the powder is shown below (usually within 2 repetitions)



Figure 5: Polyethylene powder color comparison after conditioning the sample jar (left: original Polyethylene pellets, right: powder after grinding)

Operation

(1) Cut sample < 8 mm in all dimensions and use tweezers to load sample into the sample jar

(2) Load a 9.6 mm stainless steel grinding ball into the sample jar and tighten the cap by two 9/16" wrenches

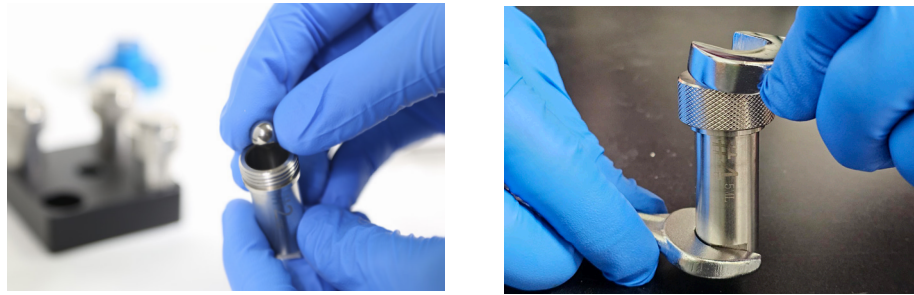


Figure 6: Load grind ball and tighten the cap

Caution

Unbalanced sample rack leads to severe damages to shaft

Use extreme caution & protecting equipment when handling liquid nitrogen

(3) Finish all 4 sample jars. All 4 sample jars need to be loaded into the sample rack to balance weight, even when not all jars are used for samples. For jars that do not have samples, the jar could be left empty without grinding balls.

(4) Pour 5 L liquid nitrogen into the supplier reservoir from a low pressure LN2 tank. It would be sufficient to last 2-3 hours.

(5) Use the supplied tongs to load sample rack with 4 jars to immerse into the liquid nitrogen for 5 minutes

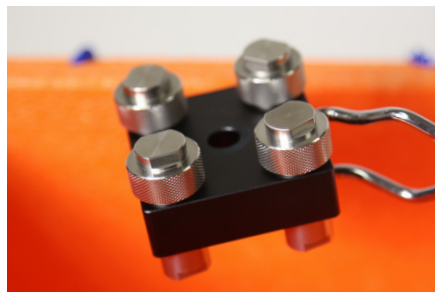


Figure 7: Immerse the sample rack with four sample jars into liquid nitrogen

(6) Use the tongs to retrieve the sample rack from liquid nitrogen and slide onto the center shaft. Please pay attention to the cavity on the bottom of the sample rack to match the sample rack up and down direction.

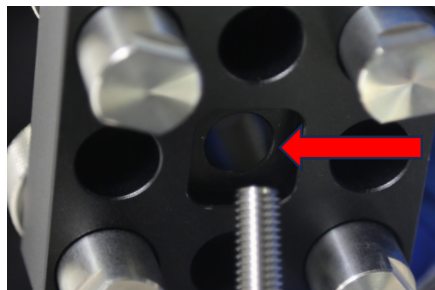


Figure 8: Slide on the sample rack

(7) Wear thermal gloves, and slide the rack cover (Figure 9) through the center shaft. Then tighten the fasten nut. Please pay attention to the direction of the fasten nut. The larger part of the fasten nut should be facing down.

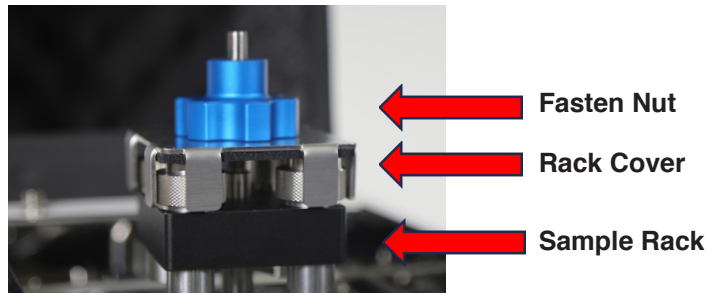


Figure 8: Sample rack cover on and tighten the fasten nut until finger tight

(8) Close the sample bay cover and lock the latch.

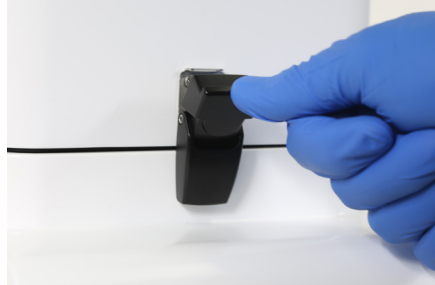


Figure 9: Lock the sample bay latch

(9) After powering on the cryo-mill, click login from the Welcome screen. No login credential is needed.

(10) A grinding method for polymers is loaded as default method #1 at 65Hz with 8 repeating runs.

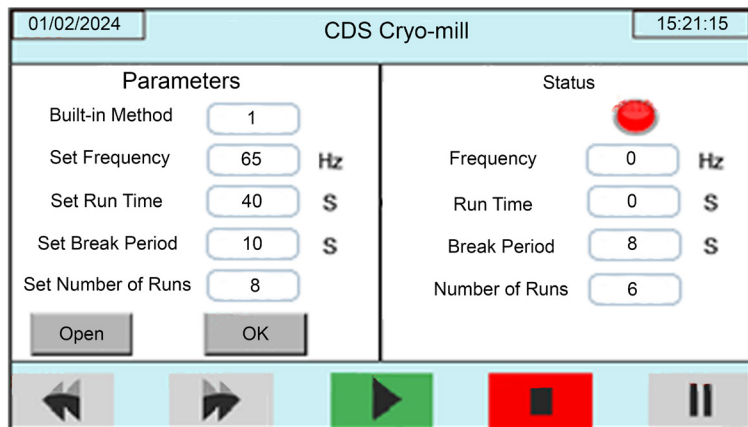







Figure 10: Main interface of the cryo-mill control

The left corner shows the date, and the right corner is the time. The left column lists parameters for the current method and the right column is the real time reading for each parameter.

On the bottom of the control screen are series of buttons.  and  buttons are quick switch buttons between multiple built-in methods. To execute a method, click the Run button . To abort the current run, click . To pause and continue the current run in a later time, click . The status light has three status: Green means the cryo-mill is running, Red means the cryo-mill is stopped, and Yellow means the cryo-mill is paused and need to click the Pause button again to resume.

(11) To edit or add the built-in method, Click the Open button  under Parameters.









Method Number	Run Time/s	Break Period/s	Number of Runs	Frequency	Confirm
1	40	10	8	65	
					
					
					
					

Figure 11: Method editor interface

The method number is a sequential integer to assist the user to quickly call out the method. Run Time is the milling time in seconds. The Break Periods is the break time during each grinding cycle to allow the user to pull the sample rack and cool in liquid nitrogen. This number is recommended to be under 30 seconds to preserve energy. For long break time, always use the Pause button . The frequency is to control the shaking frequency of the driving shaft in Hz. Confirm button needs to be clicked once so all the parameters are confirmed. The Confirm button will light up after clicking. Then click the Exit button to return to the main interface, where the OK button  needs to be clicked to update the method.

(12) After finishing the current grinding cycle and click Pause button . The status light will turn yellow.

(13) Wear thermal gloves to unscrew the Fasten nut, and then remove the Rack cover. Use the tongs to remove the sample rack with 4 jars from the center shaft, and then immerse into liquid nitrogen for 5 minutes.

(14) Repeat Step 6-8 to load sample rack into the cryo-mill.

(15) Click the Pause button  to complete the next grinding cycle. The Number of Runs will count down to assist completing the full grinding process.

Performance

Table 1 lists a reproducibility study of six polymer samples after finishing 4 and 8 grinding cycles from the default method for microplastic. The sample is then loaded into a CDS Pyroprobe for RSD comparison. In general, 4 grinding cycles would ensure a RSD of 10% and 8 grinding cycles would improve the RSD under 5% for majority of polymers.

Table 1: Cryo-mill RSD comparison

	RSD after 4 runs	RSD after 8 runs
PMMA	4.98%	3.12%
PS	3.47%	3.44%
PP	5.13%	3.84%
PVC	5.34%	4.12%
PE	10.12%	3.40%
PET	10.52%	4.19%