CDS Cryo-mill Manual

CDS Analytical
A LabTech Company

Cryo-mill for Polymers

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.

Hardware

The Cryo-mill is composed of the following hardwares as shown in Figure 1
(1) Cryo-mill
(2) Liquid nitrogen reservoir
(3) Metal tongs
(4) Touch screen pen
(5) Power adapter (Only for 110V version)

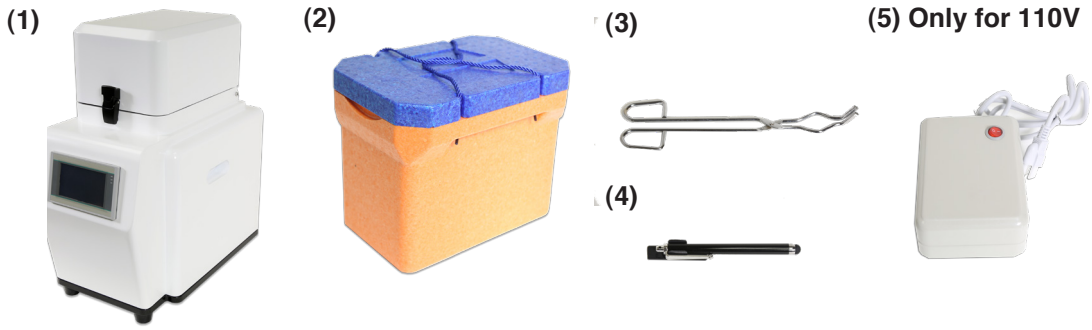

Figure 1: Cryo-mill hardware

The 110V version of the Cryo-mill comes with a power adapter. The power cord of 110V cryo-mill needs to be plugged into the power adapter, and then the power adapter needs to be plugged into the power outlet.

Four 5 ml capacity 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 amounted 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, and dispersing polymer samples.

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

Warning

Sever damages occurs without removing transportation bolts

(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.

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.

Operations

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

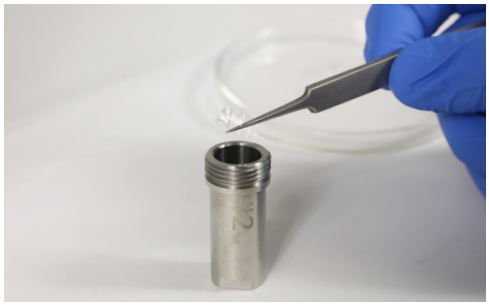

Figure 5: Cut sample to smaller pieces (<8 mm) and load into sample jar

(2) Load a 9.6 mm stainless steel grinding ball into the sample jar and tighten the cap by hand

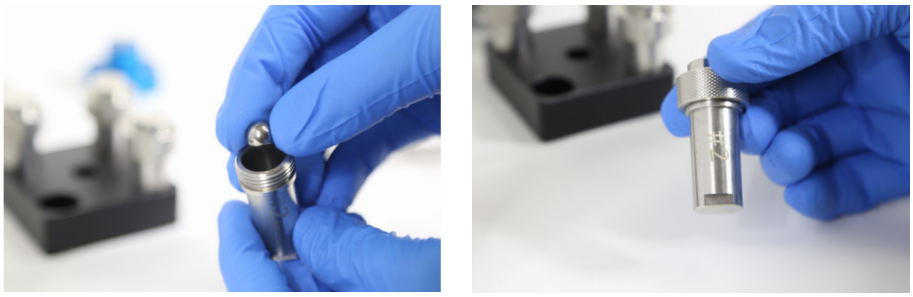

Figure 6: Load grind ball and tighten the cap

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

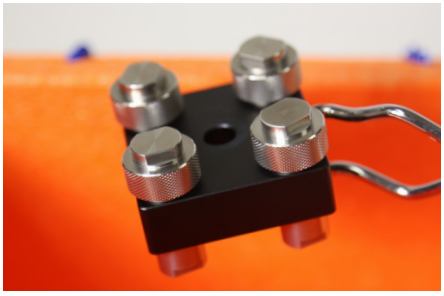

Figure 7: Immerse the sample rack with four sample jars into liquid nitrogen

(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.

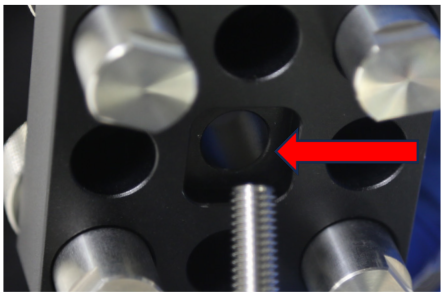

Figure 8: Slide on the sample rack

(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.

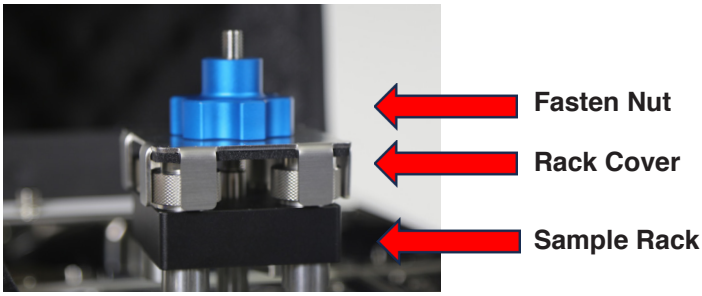

Figure 8: Sample rack cover on and tighten the fasten nut until finger tight

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

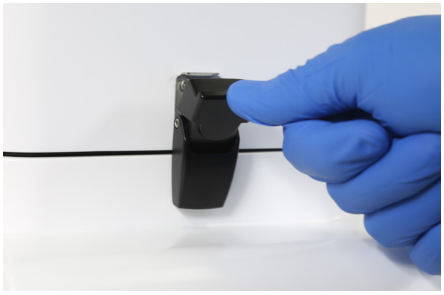

Figure 9: Lock the sample bay latch

(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 at 65Hz with 8 repeating runs.

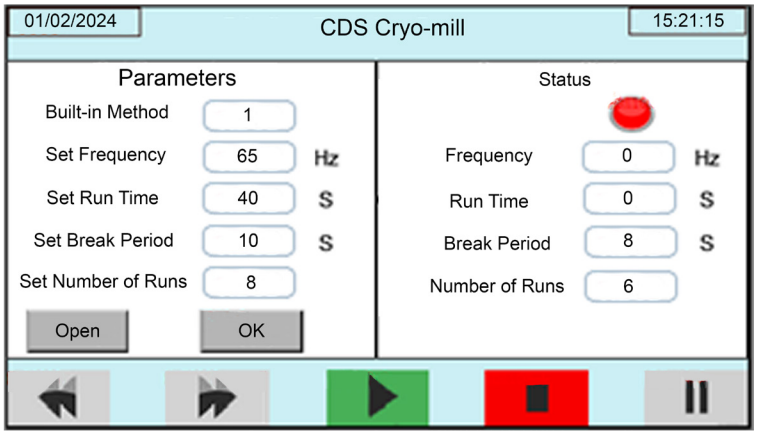

Figure 10: Main interface of the cryo-mill control

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 Run button to resume.

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

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 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 OK needs to be clicked to update the method.

(12) After finishing the current grinding cycle, wait for the status light to turn red, 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 Run 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%