

SPEX® SamplePrep®

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6875 FREEZER/MILL®



OPERATING MANUAL

For 6875-115 and 6875-230 Freezer/Mills this manual is part number 87051

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1.0 INTRODUCTION

SPEX SamplePrep Freezer/Mills are laboratory mills that cool unconventional materials to cryogenic temperatures and pulverize them to a powder form without thermal degradation. Our proven technology uses a dual electromagnetic, grinding chamber that rapidly drives a steel impactor back and forth against the two end plugs of the sample vial. Since the vial is securely closed the integrity of its contents is maintained, hazardous or critical samples are easily controlled, and cross-sample contamination is eliminated. The sample chemical composition is preserved as a result of the vials being immersed in liquid nitrogen at cryogenic temperatures throughout the grinding process. These unique aspects have made SPEX SamplePrep Freezer/Mills the most effective cryogenic mills in the world. They are the “mills of first choice” for many abnormal materials, or samples whose composition or structure cannot be ground using conventional grinding methods.

NOTE: Please do not operate the 6875 Freezer/Mill® until you have read these instructions and are familiar with its controls and operation. The Freezer/Mill is different in its principles and operation from any other laboratory mill.

OPERATING CONDITIONS: Do not set up the 6875 Freezer/Mill® in an insulated or confined space. The Freezer/Mill should be run on an open countertop, in ambient air, with recommended maximum relative room humidity 70% from 5°C - 31°C (40°F - 88°F). If the entire mill is chilled during operation, components such as the gas cylinders, controller, and display screen can fail. This damage is not covered by the Freezer/Mill warranty. During operation, water vapor in the air will condense on parts of the unit. For this reason, it is important not to place any moisture-sensitive equipment near the Freezer/Mill.

VERY IMPORTANT: Liquid nitrogen not only makes samples brittle through severe chilling, making them “grindable,” but also cools the magnetic coil which powers the 6875 Freezer/Mill. If the mill is operated without liquid nitrogen for a period of about one minute, the coil will become very hot and may sustain permanent damage. The 6875 Freezer/Mill has a liquid nitrogen sensor that should shut down the mill when the liquid nitrogen gets too low to cool the coil. Nevertheless the nitrogen level should be visually checked during extended runs as well as topped off before every run. The Freezer/Mill warranty does not cover damage to the coil caused by operating the mill with little or no liquid nitrogen.

SAFETY: Liquid nitrogen (LN) can be hazardous. Its boiling point is -195.8°C (-320.4°F). When working with liquid nitrogen directly or indirectly, the LN Tank valve or hose, or chilled Freezer/Mill components, cryogenic gloves must be worn to protect hands. Safety goggles are also recommend to protect eyes from possible splashing. Be careful not to splash liquid nitrogen onto clothes or unprotected skin. Additional information on LN safety can be found at <http://engineering.dartmouth.edu/microengineering/ln2.html>.

2.0 6875 FREEZER/MILL SPECIFICATIONS

Type of Mill:	Cryogenic impact mill
Controller:	Touchscreen
Data Transfer:	USB Port (back of controller)
Grinding Mechanism:	Steel impactor driven by dual electromagnets
Coolant:	Liquid nitrogen (LN)
Dimensions:	20½ in. (52 cm) x 21½ in. (55 cm) x 18 in. (46 cm)
Weight:	(empty, without vial or coolant) approx. 44 lbs. (20 Kg)
Grinding Vials:	One Large Vial (6801), one Mid-Size Vial (6871, 6881, 6885), one to four Small Vials (6751, 6761, 6771, 6781S) or one Microvial set (6757).

Typical Vial Capacity: ***Small Vials:*** actual volume with impactor, approx. 25 ml. Typical sample weights: 2 grams for biological samples, 1 gram for polymers.
6757 Microvial Set: 100 - 500 mg per individual 6757V Microvial, depending on sample.
Mid-Size Vials: up to 5 times the sample capacity of Small Vials.
Large Vials: Approximately twice the sample capacity of Mid-Size Vials, or up to 10 times that of Small Vials.
 Actual performance of any vial/sample combination depends on sample properties, cooling time, grinding time, desired outcome, etc.
 Effective capacity for a given sample is determined by experiment.
 Overfilling a vial greatly reduces its efficiency.

Typical Liquid Nitrogen Consumption: 10-15 liters for initial cool-down and filling of the tub.
 4-6 liters for each hour of operation.
 Actual LN consumption can vary.

Electrical Specifications: CE Approved. Available as 115V/60HZ or 230/50HZ.
Fuses: Two 15-amp slow-blow fuses in AC Input module.
 25-amp slow-blow fuse on driver board.
Power Cord: 115V/60HZ version: 3-prong grounded plug supplied.
 230V/50HZ version: 2-prong European plug supplied.
Operator is responsible for supplying alternate line cord/plug. (If necessary)

Safety Features: Lid Interlock prevents mill from running if lid is not latched shut.
 Liquid nitrogen sensor shuts down mill if LN level is too low.

NOTE: *The sensor can shut down the mill during a run, so the liquid nitrogen level should be visually checked during extended runs as well as before every run. The Freezer/Mill warranty does not cover damage to the coil caused by operating the mill with little or no liquid nitrogen.*

3.0 UNPACKING

Inspect the exterior of the packing box and report any visible damage to the carrier. Remove all packing documents from the exterior of the box, and save them for your records. Open the top of the shipping box. Remove the packing material and accessories, and gently remove the 6875 Freezer/Mill. Visually check the mill for any damage that may have occurred during shipping. Unlatch the lid and inspect the interior of the tub, ensuring that it is free of any packing material. Check the packing list to see that there are no parts missing, and inspect the accessories. We recommend storing the packaging materials in the event there is a need to return the unit for warranty service or repairs.

Each new 6875 Freezer/Mill® is supplied with a 6804 Extractor and a 6805 Vial Rack. Grinding vials are necessary for processing samples but must be purchased separately. The full range of grinding vials and accessories for the 6875 Freezer/Mill is described in our catalog, the Handbook of Sample Preparation and Handling, and at www.spexsampleprep.com.

Inspect the accessories. Check the packing list to see that there are no parts missing. If everything seems to be in proper order, store the packaging materials, in case there is a need to return items.

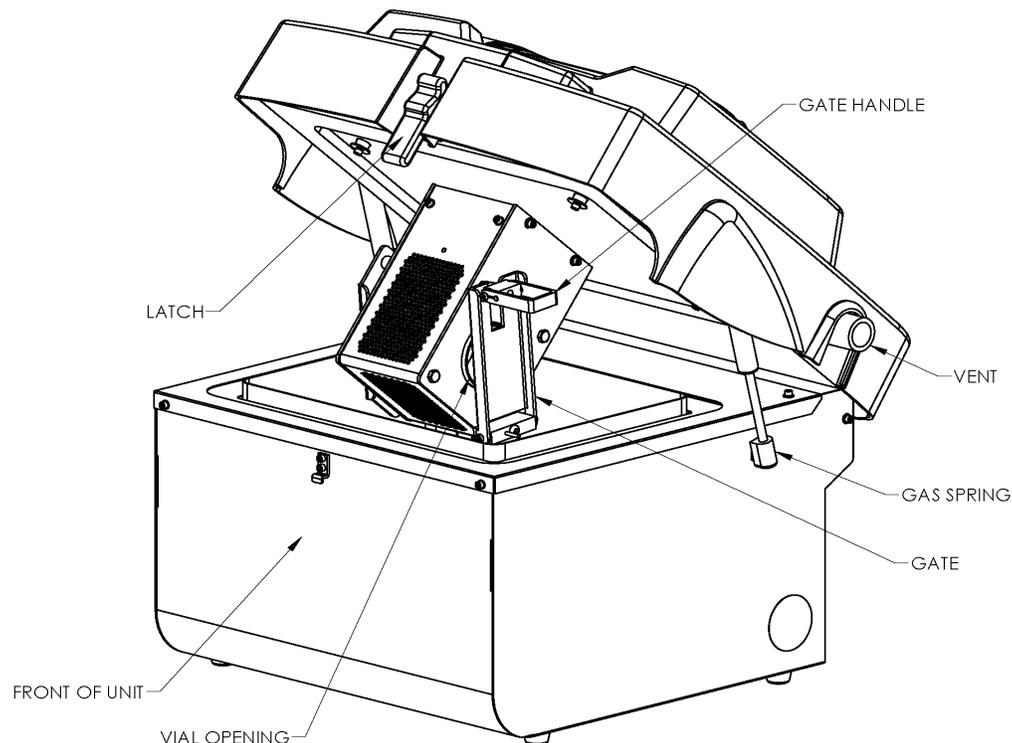


Figure 1 - 6875 Freezer/Mill, Front View

3.0 UNPACKING (Cont'd)

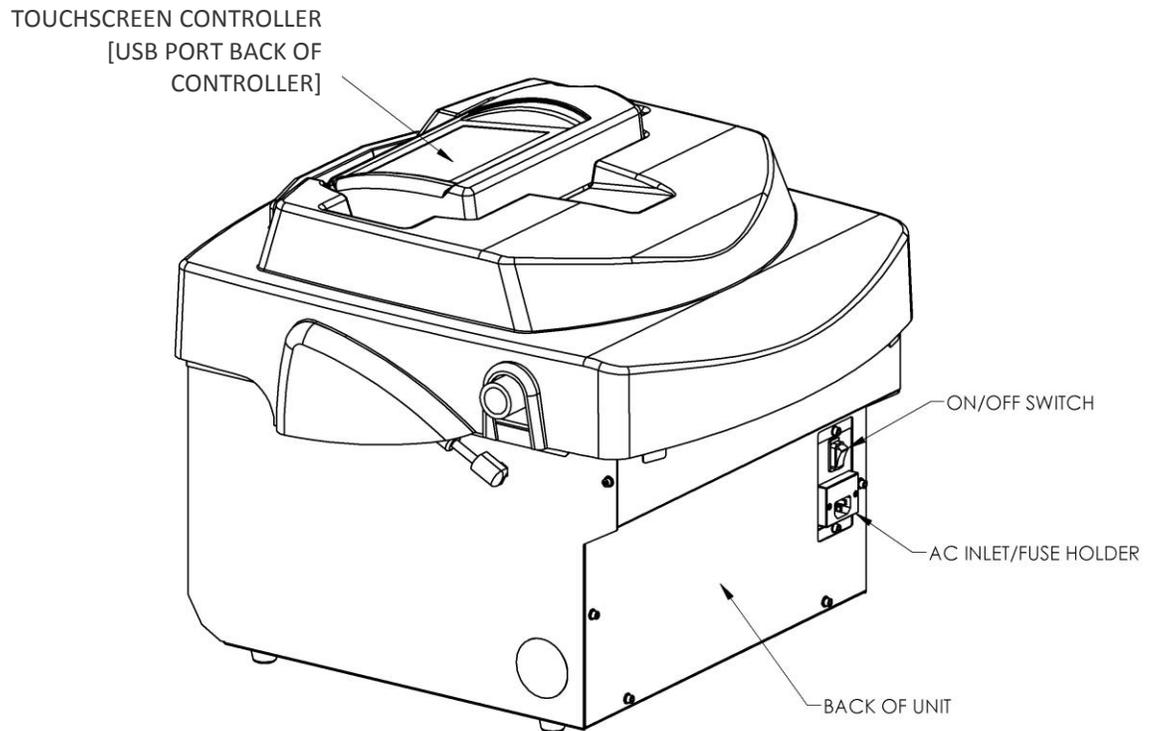


Figure 2 – 6875 Freezer/Mill, Rear View

4.0 SETTING UP

The 6875 Freezer/Mill weighs 44 pounds (20 Kg) empty. The lid opens from the front and the lid latch is in the center. The AC Input module is on the back of the cabinet, and includes the power switch and fuse tray. The fuse tray can be opened with small screwdriver or knife-blade. Also on the back of the cabinet is the vent from the liquid nitrogen tub. There are gas cylinders on both sides of the cabinet to control the movement of the lid. The controller is affixed to the top of the lid and linked to the unit by a RJ45 Ethernet cable connector. At the back of the controller is an USB Port to upload and download data, or to upgrade software.

4.1 Electrical Connection

Plug the electrical cord into the inlet on the back of the Freezer/Mill cabinet, and then into a standard 3-prong grounded electrical outlet. A fused 20-amp circuit is recommended for the 115V/60 HZ version. The 230V/50HZ, 6875 Freezer/Mill is supplied with a standard European 2-prong plug with cord. For the 230V/50HZ version, make sure the cord and plug conform to local electrical codes.

The fuses are next to the electrical cord inlet in the Fuse Holder. To replace a fuse, first remove the electrical cord from the inlet. Use a small screwdriver to pry open the fuse panel. There are two individual fuse holders, each with a protruding tab. Push the tab sideways to pop out the fuse holder allowing the holder to be removed with the fingers. **Never use pliers or force to remove a fuse holder.** Both fuses must be replaced. (Section 10.1)

4.2 Power Switch and Controller

Electric power to the Freezer/Mill is controlled by a rocker switch on the inlet module. It is marked with two numerical symbols (0 for OFF and I for ON). Press the “I” side of the switch to turn the power ON, or press the “0” side of the switch to turn the power OFF. When power to the mill is turned ON, the controller display will light up.

The controller screen is a touchscreen that can be activated by a fingertip or stylus. Data is transferred to other computer devices via USB Port, located in back of the controller. The 6875 Freezer/Mill has one grinding chamber, which is programmed on the Control Panel screen (Section 5.2). The programmable parameters include Cycles (number of grinding periods), Precool Time (initial chilling of sample), Run Time (grinding period), Cool Time (time between grinding periods), and Rate (speed of the impactor in cycles per second). The Control Panel also displays a warning if the lid is not fully closed, or if there is not enough liquid nitrogen in the tub.

The viewing angle of the controller on the 6875 Freezer/Mill can be adjusted to minimize glare. To adjust the viewing angle, simply grasp the back of the controller box and gently pull it forward until the desired angle is reached, as shown in Figure 3. If it is necessary to send the unit back to SPEX SamplePrep for service, be sure to return the controller to its horizontal position before packing the unit for shipping.

4.2 Power Switch and Controller (Cont'd)

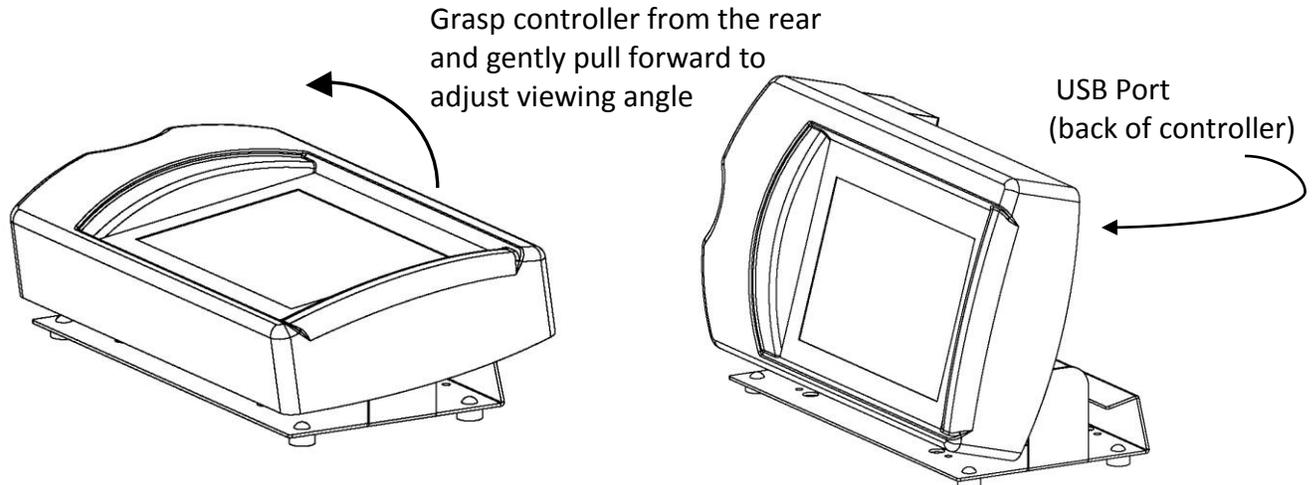


Figure 3 - Adjusting the Viewing Angle of the Controller

4.3 Lid, Coil Assembly, and LN Sensor

The lid of the 6875 Freezer/Mill supports the coil (dual electromagnet assembly) and coil housing. To open the lid, hold the lid down with one hand, and with the other pull the upper end of the latch toward you. The lower end of the latch should disengage from the cabinet. Release the lid and it should be pushed up by the gas cylinders, which also keep the lid raised when open. The lid must be open for insertion and removal of vials. Liquid nitrogen (LN) is added with the lid open, unless the 6875 is equipped with the 6820 Auto-Fill System (Section 7.0).

A vial is inserted into the circular hole on the right side of the chamber housing. The gate keeps the vial or vials in place during grinding, but must be lowered to insert or remove vials. **Do not run the 6875 Freezer/Mill without a loaded vial in the chamber.**

The lid must be shut and latched for the Freezer/Mill to run. Push the lid down with one hand, and hold it down while engaging and closing the latch.

The chamber gate (Figure 1) holds the vial or vials in the chamber during grinding. To lock a vial in place, loosen (unscrew) the chamber gate handle a turn or two, and position it so the handle passes through the slot in the chamber gate when the gate is raised. Tighten the chamber gate handle against the gate, so the gate contacts the vial firmly, but **do not overtighten the gate** as this can crack the plastic center section of the vial. When the chamber gate handle is properly tightened, fold it down to lock the gate during grinding. Always use the proper Adapters for Mid-Size and Small vials (see Section 4.4), and **always wear gloves when touching a chilled vial or any part of the chilled chamber housing!**

4.3 Lid, Coil Assembly, and LN Sensor (Cont'd)

The liquid nitrogen (LN) sensor is the small tube mounted on the left wall of the LN tub. If there is not enough liquid nitrogen in the tub to start the mill when the  button is touched, **LN LEVEL LOW** screen (Section 5.4) will display LN LEVEL LOW. If the LN runs low during a grinding program the same message will appear and the program will stop. To continue running, fill the tub with liquid nitrogen to the mark on the back of the tub. When the  button is pressed, the grinding program will begin where it left off.

4.4 Grinding Vial Sizes, Adapters, and Vial Openers

Large vials (6801, 6871) are run one at a time in the 6875 Freezer/Mill, without any adapter. Large vials are opened using the 6804 Extractor/Vial Opener, which is part of the 6870L Accessory Package or the optional 6808 Large Vial Extractor.

Mid-Size vials (6881, 6883, 6885) are run one at a time, but the 6887 (6888) Mid-Size Adapter must be placed in the chamber first, to center and align any Mid-Size vial. Mid-Size vials can be opened by the 6804 Extractor/Vial Opener, but only by installing the 6884 Mid-Size Vial Adapter for the 6804. The 6884 and 6887 Adapters comprise the 6870M Accessory Package.

Most Small vials (6751, 6761, and 6771) can be run one, two, three, or four at a time in the 6875 Freezer/Mill. However, they must be used in conjunction with the 6807 Multi-Vial Holder. The 6807 Adapter keeps the vials parallel to each other, so each impactor moves back and forth in line with the magnetic field. When running Small vials, make sure the chamber gate contacts the end plug of every vial. If the gate needs adjustment to do this, the bottom or pivot end of the gate can be moved toward or away from the chamber housing by turning the gate clockwise or counterclockwise on its mounting bolt.

5.0 TOUCHSCREEN DISPLAY

The 6875 Freezer/Mill is programmed and operated through a series of touchscreen displays. Transition between screens, and all programming and operating commands, are done by touching the screen with a fingertip or stylus. ***Do not use a sharp point as this can damage or deface the screen.***

5.1 Logo Screen

When the power is switched ON at the back of the mill, "Please wait" appears during start-up as the software loads. Then the SPEX SamplePrep logo is displayed, with a web address and a brief animated representation of the Freezer/Mill. After the animation has finished, the screen will switch to the **HOME** screen, as shown in Figure 4. From the Home screen the Control Panel, Saved Protocols, and Resources (Run History, Tech Support, Accessories, Training material) can be accessed by touching the buttons displayed.

5.1 Logo Screen (Cont'd)

The Home Screen  Icon can be found on the following screens (e.g. Control Panel, Resources, Saved Protocols) positioned to the bottom right of the screen. Touching the Home Icon allows the users to return to Home Screen.



Figure 4 – Home Screen

5.2 Control Panel

NOTE: To keep a run from being interrupted by the liquid nitrogen (LN) sensor, the LN level should be visually checked from time to time during extended runs, as well as before every run.

The Control Panel displays the programmed run parameters, as shown in Figure 5. Changes to the run settings, are made from this screen. The 6875 Freezer/Mill is **Only** equipped with one Grinding Chamber.

To recall stored run protocols, touch the  button located at the bottom right of the screen. (See section 5.4 for more information on Saved Protocols)

--To return to the Home Screen touch the Home Icon. 

5.2 Control Panel (Cont'd)

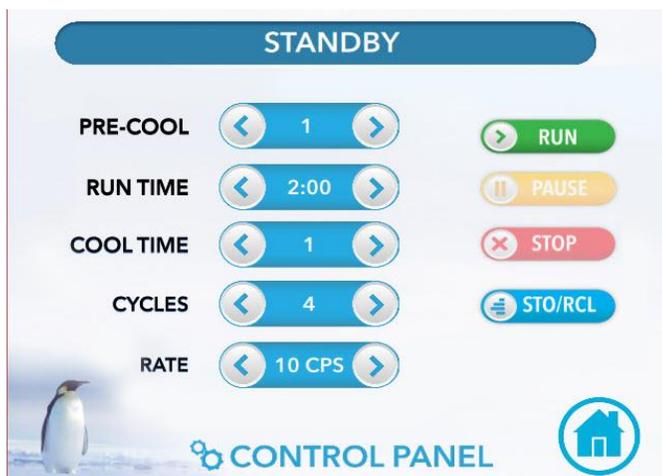


Figure 5 – Control Panel

The following sequence of events will occur from the current settings in Figure 5

1. Precool - the coil is inactive while the vial cools in liquid nitrogen for 1 minute. Samples must be precooled before grinding (5 to 10 minutes is recommended).
2. Cycle 1 - the coil is activated and the impactor grinds for 2 minutes at a rate of 10 cycles per second (20 impacts per second).
3. Cool Time - the coil is inactive for 1 minute between grinding cycles allowing coil and sample vial to cool down.
4. Cycle 2 - the coil is activated and the sample is ground for another 2 minutes.
5. Cool Time - the coil is inactive for 1 minute, as in step 3.
6. Cycle 3 - the coil is activated and the sample is ground for another 2 minutes.
7. Cool Time - the coil is inactive for 1 minute, as in step 5.
8. Cycle 4 – the coil is activated for the final 2 minute cycle. At the end of the final cycle the program is complete and the sample has been ground for a total of 8 minutes.

In the **CONTROL PANEL**, The STATUS BAR (top of screen) counts down the Time Remaining for a run. Before a run starts, the STATUS BAR will display STANDBY, as shown in Figure 5.

To change the settings of a selected field, press the arrow buttons on the **CONTROL PANEL** screen. The left arrow buttons decrease the number displayed and the right arrow buttons increase the number displayed.

5.2.1 Starting a Programmed Run

To run the program displayed on the **CONTROL PANEL** screen, touch  button. The Freezer/Mill can be started, stopped, or paused in the middle of a grinding program from the button selections to the right of the screen.

Open the lid and place the loaded sample vial in Coil Vial Opening, then **SLOWLY** close the lid to keep the liquid nitrogen from splashing or spilling out the tub. Two additional loaded sample vials can be placed in the precooling basket while running sample. By precooling the additional loaded sample vials, the PRECOOL time for these samples can be decreased or eliminated during the next run. After all vials have been loaded, lower the lid and secure the latch. If the liquid nitrogen in the tub is not at the correct operating level, then the screen will display LN LEVEL LOW (Figure 6). Add more LN and touch RESUME to begin operation.



Figure 6 – LN Level Low Screen

If the lid is not completely closed and latched, the screen will display LID OPEN, as shown in Figure 7. To abort the program touch the  button. To continue with the program touch the RESUME button after the lid has been completely closed. When the tub is filled with liquid nitrogen to the correct level, and the lid is securely latched, the STATUS BAR will turn **green** and begin counting down. The PRECOOL sequence will start indicating the beginning of the grinding program.

5.2 Control Panel (Cont'd)



Figure 7 – Lid Open Screen

During the PRECOOL stage the TIME REMAINING line counts down the time for that stage in 1-second increments. To pause the program, touch the **PAUSE** button. To continue the program touch the RESUME button. To abort the program, touch the **STOP** button. To start a new program, touch the **RUN** button. When the programmed stage (e.g. PRECOOL) is complete a **green check mark** will appear next to that field. (Samples must be precooled)

Following the PRECOOL stage is the first GRINDING cycle stage. The RUN TIME line will display the time remaining in that cycle of the grinding stage. At the end of each grinding period a **green check mark** is placed next to that field. If there are 4 total grinding periods, the CYCLES line will countdown to “0”.

NOTE: By touching the Run screen (and hold), during a programmed run displays the current run settings in an **Orange Color Font**.

The protocol description located bottom of screen will change to **SETTINGS**.



Figure 8 – Run Screen during grinding

5.2 Control Panel (Cont'd)

As shown in Figure 8, the RUN TIME line indicates the 2 minute grinding period has been completed for the 1st grinding Cycle out of 4 grinding Cycles. Between any two grinding periods is the COOL DOWN stage. The COOL TIME line display, the time remaining in the COOL DOWN stage. The Cool Time for 1 minute is set to start as indicated by the yellow symbol. At the end of each Cool Time period a **green check mark** is placed next to that field. The Rate is 10 cps (cycles per second).

The STATUS BAR (top of screen) counts down the Time Remaining for the programmed run. The Time Remaining to complete the programmed run is 11 minutes and 58 seconds, as displayed in Figure 8. The bottom of the screen displays the Protocol Name for the run. If no Protocol Name is assigned to the run as a Saved Protocol (Section 5.4) then the default name will be displayed as COUNTDOWN. When the grinding program has ended, the screen will display RUN COMPLETE (Figure 9). Touch the screen to return to the Control Panel.

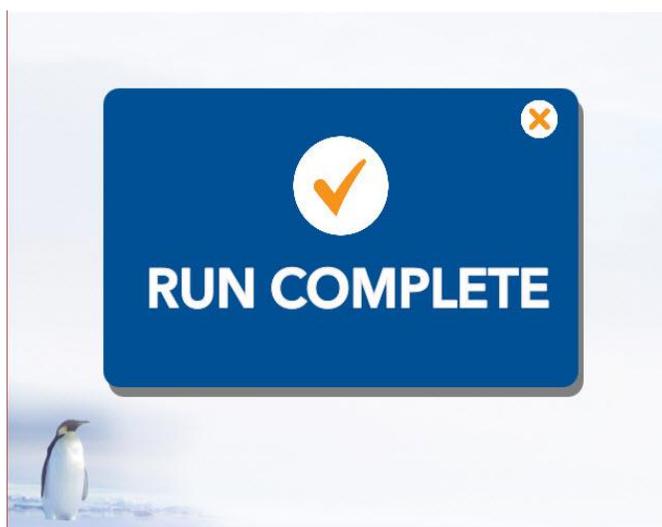


Figure 9 – Run Complete Screen

5.2.2 Adjusting the Grinding Rate

While in the **CONTROL PANEL** screen, the only parameter that can be modified during grinding is the RATE. It is adjusted by touching the right and left arrow buttons next to the Rate field. Touching the right arrow increases the rate by 1 cps at a time, while touching the left arrow decreases the rate by 1 cps. Tap or hold the right or left arrow buttons until the desired rate is reached.

Rate is the number of back-and-forth cycles per second (cps) completed by the impactor. During one cycle the impactor strikes both end plugs of the vial, therefore a rate of 10 cps is equivalent to 20 impacts per second. The factory default setting for the rate is 10 cps. The maximum grinding rate is 15 cps and the minimum is 5 cps.

5.2.3 Stopping or Pausing a Run

To stop a program, touch the  button on the **CONTROL PANEL** screen and the program will end. If the  button is touched after the program has stopped then the entire program will start over.

To pause during a grinding program, touch the  button. The **RUN** will hold the settings at that moment (elapsed time, cycle, etc.). To resume the program, touch the Resume button. This will restart the program at the point at which it was paused. Alternatively, pressing STOP will end the program.

5.3 Changing Settings

To change parameters (precool time, grinding time, cool down time, cycles and rate), touch the arrow buttons on the **CONTROL PANEL** screen. The changes will appear in the blue area between the arrows, as shown in Figure 10.

The CONTROL PANEL allows the user to change the parameters by touching the right and left arrows next to the field. Touching the right arrow increases the parameter, while touching the left arrow decreases the parameter. Tap or hold the right or left arrow buttons until the desired number is reached. The arrow key will turn blue when the maximum or minimum number is exceeded. To run a program with the new settings, touch the  button.

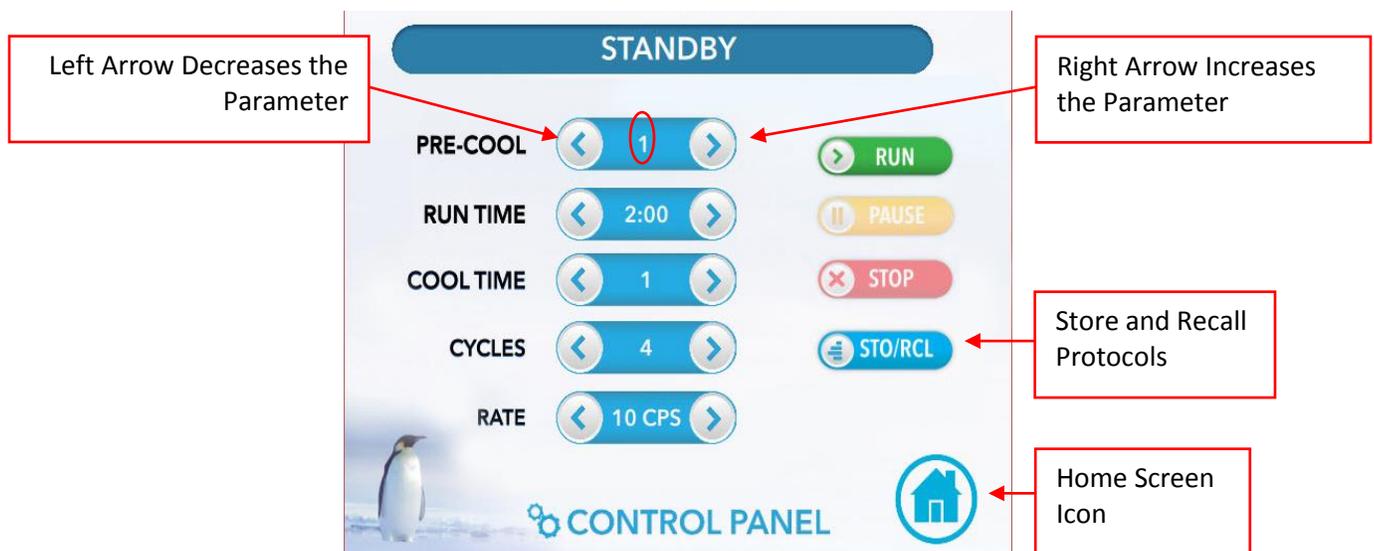


Figure 10 – Changing Settings

5.4 Saved Protocols

The **SAVED PROTOCOLS** screen is shown in Figure 11. In this example, three saved protocols have been stored and named (e.g. Hair, Bone, Soft Tissue). Up to 20 Protocols can be stored. Only 10 protocols at one-time are visible on the Saved Protocols screen. To access or make visible additional protocols, or vacant protocol spaces (e.g. User Protocol) swipe up or down the touchscreen. This brings up vacant protocol slots on the **SAVED PROTOCOLS** screen.

A saved program retains the settings for number of cycles, precool time, run time, and cool time between cycles. Be sure to adjust the rate to the desired setting after recalling a stored program.

The Default protocol recalls the last programmed run setting that was not saved. The Default protocol can be changed without saving the new settings, as described in section 5.4.

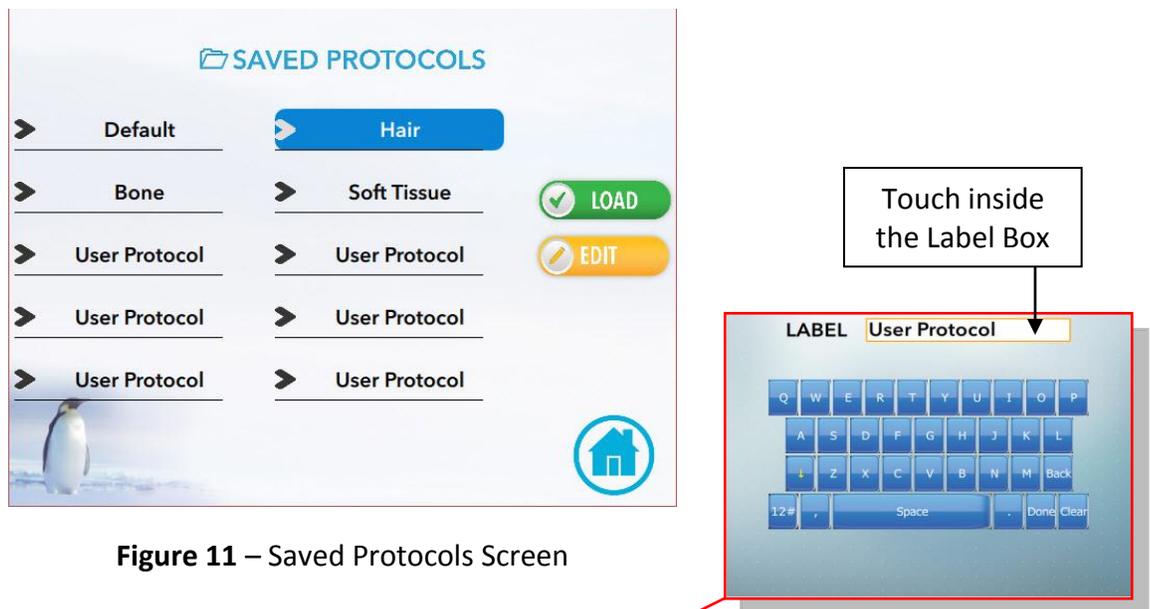


Figure 11 – Saved Protocols Screen

5.4.1 Storing a new program

To store the new program or to recall a stored program, touch the **STO/RCL** button on the Control Panel screen (Figure 5). In the **SAVED PROTOCOLS** screen touch inside the Label Box, as shown in Figure 12.

This will bring up the **KEYBOARD** screen, a simplified version of the standard keyboard for a computer. In addition to number and letter keys, this keyboard has standard symbol keys (#, %) and four function keys (SPACE, BACK, DONE, and CLEAR).

As the letters/numbers are touched, they appear above the keyboard in the Label Box with a centered cursor. To access the number and symbol keys touch the 12# button located bottom left on the **KEYBOARD** screen. To switch back to letter keys touch the ABC button (same button) bottom left on the **KEYBOARD** screen.

5.4 Saved Protocols (Cont'd)

The Up/Down yellow arrow key on **KEYBOARD** allows the user to shift back and forth from upper case to lower case (the default is upper case). Touch Down arrow to switch to lower case. Or touch Up arrow to revert to upper case.

Touching the SPACE key advances the cursor one space. Touching the BACK key deletes one space. The CLEAR key deletes whatever has been entered in the Label Box.

Touch the DONE button to save the name shown in the Label Box. The label will appear as the name of the program in the **SAVED PROTOCOLS** screen (Figure 11).

The CANCEL key does not change anything on the screen, but returns the display to the **SAVED PROTOCOLS** screen.

To run the newly saved protocol, touch the protocol to highlight (**BLUE**) the name. Then touch the LOAD button to send protocol to the **CONTROL PANEL** screen. Review the parameters and touch the **RUN** button to initiate the protocol.

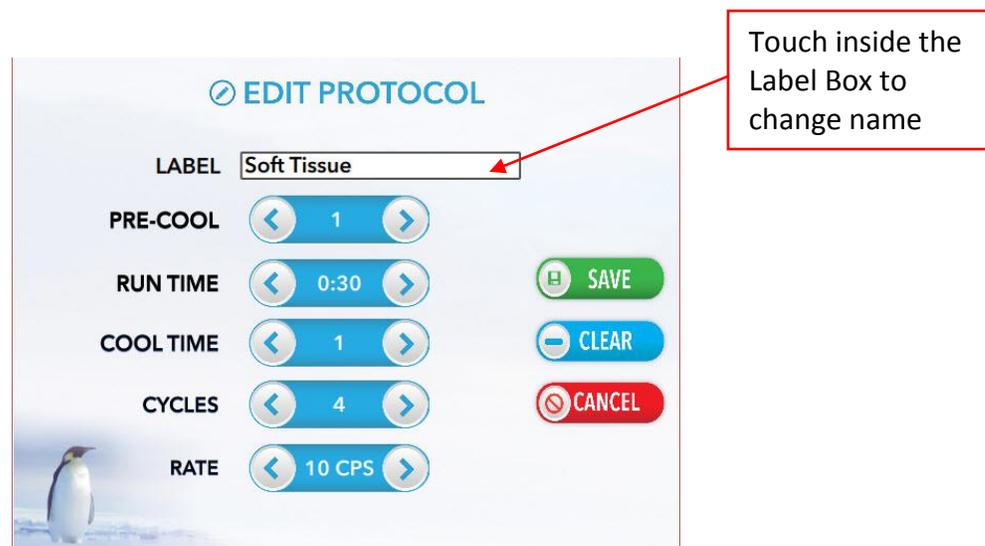


Figure 12 – Edit Protocol Screen

5.4.2 Delete Protocol

To delete a saved protocol, touch the protocol to highlight (**BLUE**) the name. Then touch the EDIT button. From the Edit Protocol screen touch the CLEAR button. A pop-up window opens confirming to delete this protocol, as displayed in Figure 13. Touch YES to clear or touch NO to keep the saved protocol. To rescind this action touch CANCEL button. To retain the action, touch SAVE button. Saving the clear protocol returns the parameters to the default setting (i.e. User Protocol).

5.4 Saved Protocols (Cont'd)

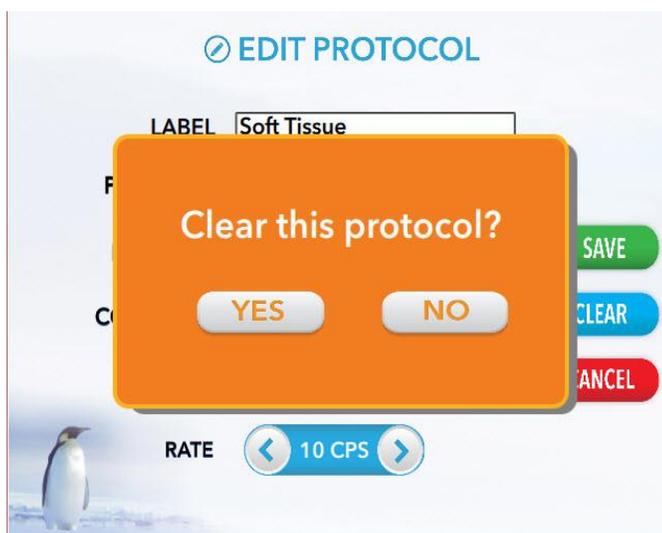


Figure 13 – Clear Protocol Screen

5.4.3 Edit Saved Protocols

To edit a saved protocol, touch the protocol to highlight the name (BLUE). Then touch the EDIT button. From the Edit Protocol screen change parameters and/or name, then touch the DONE button.

5.4.4 Recalling a program

In the **SAVED PROTOCOLS** screen, touch the protocol to highlight the name (BLUE). Then touch the LOAD button to send protocol to the **CONTROL PANEL** screen. Review the parameters. To RUN the protocol touch the  RUN button to initiate the protocol.

6.0 OPERATION

6.1 Adding Liquid Nitrogen Coolant

Liquid nitrogen is added to the 6875 Freezer/Mill with the lid open. The 6875 should be filled until the LN level reaches approximately 2 inches (5.1 cm) below the lip of the tub. If the liquid nitrogen is above this level when the lid is closed, LN can splash outside the tub. **Always close the lid slowly to avoid splashing.**

Liquid nitrogen will most likely be available either in a large tank with a flexible steel hose, or in smaller Dewar flasks. The 6875 Freezer/Mill typically consumes 10 to 15 liters of LN during initial cool-down, and another 4 to 6 liters per hour, depending on use.

6.1 Adding Liquid Nitrogen Coolant (Cont'd)

Unlatch the lid and the gas cylinders will push it open. To unlock the latch, press down on the lid and pull the top of the latch forward and down. Disengage the bottom of the latch and the lid can be opened. The coil assembly is attached to the lid and will rise with it. The chamber gate is on the right side of the coil housing. To lower the gate to insert a loaded vial for grinding, lift the coil gate handle to the horizontal position and twist to align with the slot in the coil gate. Then pull the coil gate to the right to open.

Note that there are three different sizes of vial that can be run in the 6875 Freezer/Mill: Large, Mid-Size, and Small. Mid-Size vials must be run with a 6886 Adapter in the coil, and Small vials must be run with a 6806 Adapter in the coil. Large vials do not need an Adapter. See Section 4.4 for details.

With the vial or vials (and adapter) in place, loosen the coil gate handle a turn or two counterclockwise, align the handle with the slot, close the gate, and turn the handle clockwise until the gate is in snug contact with the vial or vials. To lock the gate, fold down the gate handle against the gate. ***Do not overtighten the gate, as this can crack the vial's center section.***

If the Freezer/Mill tub is empty, pour liquid nitrogen (LN) into the tub until it is approximately on third full. Initially LN will boil off vigorously, but as the tub cools the boiling will subside. Add more LN gradually, to avoid splashing and boiling over, until the LN level is approximately 2 inches below the rim of the tub. When boiling is subdued, push the lid down gently until the bottom of the latch can be engaged. Pause if splashing becomes excessive. When the vapor stream has subsided, open the lid again and top off the liquid nitrogen in the tub, filling it to the mark on the inside wall of the tub. Insert a loaded vial or vials into the coil (if this has not already been done), and tighten and lock the coil gate.

Press the Freezer/Mill lid closed and latch it. When the lid is closed and latched, further nitrogen boiling will be visible as a stream of condensed water vapor ("fog") from the vent in the rear of the Freezer/Mill; this is normal. A grinding program can now be run, as outlined in Section 5.2.

6.2 Loading Samples into Freezer/Mill Vials

6.2.1 Introduction and General Recommendations

Start by choosing a sample that is typical of those to be ground on a regular basis. As a rule of thumb, the sample pieces should be small enough to circulate inside the vial as the impactor moves back and forth, and should take up no more than $\frac{1}{3}$ to $\frac{1}{2}$ the volume of the vial, with the impactor in the vial. Typical sample sizes for the different sizes of Freezer/Mill vials are shown below in Table 1.

In practice, the optimum parameters (sample weight and size, precooling time, grinding times and number of cycles, impactor rate, etc.) are customized to match particular samples.

6.2 Loading Samples into Freezer/Mill Vials (Cont'd)

Your own procedure for specific materials should be determined by trials; in practice almost no two materials grind alike.

In practice, the optimum parameters (sample weight and size, precooling time, grinding times and number of cycles, impactor rate, etc.) are customized to match particular samples. Your own procedure for your own materials should be determined by trials; in practice almost no two materials grind alike. Avoid overfilling vials as this greatly reduces grinding efficiency.

If at first the sample doesn't grind as intended, try these strategies:

- Reduce the overall weight of the sample.
- Reduce the size of the individual pieces to ¼ or 1/8 inch (6 to 3 mm).
- Increase the precooling time. (20 minutes of precooling is about the maximum necessary for most samples to reach LN temperatures.)
- Grind longer. (30 minutes of actual grinding time is the maximum recommended for one run, due to the LN capacity of the tub, but you can top off the LN and repeat the run if necessary.)

Table 1 - Typical Sample Sizes

Sample	Large Vial 6801, 6803, 6871	Mid-Size Vial 6881, 6883, 6885	Small Vial 6751, 6761, 6771, 6781S	Microvial 6757
Plant tissue, bone, muscle, etc.	20-50 g	5-20 g	2-5 g	100-300 mg
Polymer pellet	10-20 g	2-10 g	1-2 g	50-100 mg

6.2.2 Size of Sample Pieces

The size of sample pieces is important. For uniform results, most samples should be cut into pieces smaller than ¼ inch (6 mm). Very tough samples such as fresh bone should be cut smaller, down to ⅛ inch (3 mm). There are several reasons for this:

- The more irregular the size of the sample pieces, the more difficult it is to predict the results. Uniform procedures work better with uniform samples.
- Thin fibers and films can be very difficult to cryogrind unless they are cut into small pieces first. Wads of long fibers and crumpled balls of film take up extra room and can be elastic, even at LN temperatures.
- Tough samples in large pieces can be very difficult to grind, and in rare cases may damage the vial. A large, wedge-shaped piece of bone or a tough polymer can act as a wedge to push a moving impactor through the side of the polycarbonate tube.

6.2.3 Loading a Sample into a Vial at Room Temperature

Most Freezer/Mill samples do not have to be kept cold all the time, and can be loaded at room temperature. To load a vial, first assemble it halfway by pushing an end-plug into a center cylinder. Add the sample and impactor to the vial, and close it with the other end-plug. Shake the vial to make sure the impactor has room to move back and forth.

6.2.4 Loading a Cold Sample into a Vial and Keeping it Cold

Some samples must be kept cold, such as plant or animal tissue being prepared for RNA extraction. When a sample is cold, and must be kept cold throughout the grinding process, the loading procedure is more elaborate.

Assemble the center cylinder and blunt end plug, as in 6.2.3, then chill the end plug and about an inch of the cylinder in a shallow liquid nitrogen bath. The vial rack that comes with the 6875 Freezer/Mill can be placed in an insulated container and used to hold the vials upright during chilling. At the same time, chill the impactor separately. As soon as the submerged end of the vial is chilled, drop in the cold sample and then the chilled impactor. ***(Always wear insulated gloves when handling chilled vial components directly!)*** Now carefully insert the other end plug, still warm, into the warm end of the cylinder. The vial can now be carefully placed in the mill, or submerged in a liquid nitrogen bath outside the mill, as long as care is taken not to let the sample touch the warm end plug before it has cooled down.

CAUTION: Never force an end plug (warm or cold) into a polycarbonate cylinder (warm or cold). Polycarbonate shrinks when chilled, and can crack if stressed. If the cylinder has cooled to the point that the end plug fit is too tight, allow the cylinder to warm up before inserting the end plug.

6.2.5 Loading a Very Small Sample into a Vial and Keeping it Cold

A variant of the loading technique in Section 6.2.4 can be used to grind very small samples with the open pre-chilled vial upright. Place the sample on the chilled end plug and add a small amount of fluid such as an extraction medium or water. The frozen fluid will bulk up the sample enough so it will circulate in the vial during grinding. To speed up this process, some users first prepare small “ice cubes” of the sample frozen together with 0.5 ml to 1 ml of fluid.

6.3 Loading a Vial into the Freezer/Mill

Before loading a vial or vials in the 6875 Freezer/Mill, put on cryogenic gloves. Unlatch and lift the lid of the mill, and open the coil gate. If Small or Mid-Size vials are being used, make sure the proper Adapter is in the coil. Pick up the vial(s) with a gloved hand or an Extractor/Vial Opener and insert the vial(s) into the coil. Close the coil gate and tighten it against the vial(s) by turning the gate handle clockwise, but do not overtighten the gate. Lock the gate by folding down the handle.

Additional vials can be placed in the precooling basket and carefully immersed in the LN tub on the right hand side (the end which faces the coil gate) with the basket handle against the tub

6.3 Loading a Vial into the Freezer/Mill (Cont'd)

wall. This allows additional vials to precool during the grinding process. Thus, when the next vial is to be run, the precool step can be skipped. ***Remove the basket from the LN with caution. Do not immerse a hand into the LN, even when wearing a protective glove. Use a pair of tongs to grasp the basket handle if it is below or near the LN level.***

Check the liquid nitrogen (LN) level and top it off if necessary. Gently close the Freezer/Mill lid, and latch it shut. At this point a grinding program can be run using the controls described in Section 5.

6.4 Removing a Vial from the Freezer/Mill

At the conclusion of the grinding cycle, unlatch the lid and let the gas cylinders lift it. Do not force the lid, but allow it to rise at its own speed. If the gas cylinders are too worn or chilled to lift the lid, lift the lid gently.

Remove the vial or vials from the coil, using gloves or an Extractor/Vial Opener. At this point either close the lid to conserve LN or insert another vial and begin another grinding cycle.

6.5 Opening, Emptying, and Cleaning Freezer/Mill Vials

6.5.1 Opening a Vial

To open the large vial, slip the open end of the 6804 Extractor/Vial Opener over the flanged end-plug, align the pegs in the end plug with the slots in the Extractor, and turn the knob clockwise until the end plug is drawn out. If the end plug is drawn out within a millimeter or two of the end of the center cylinder and stops, rock the 6804 Extractor gently to dislodge the end plug. Always be careful that the vial does not fall and spill its contents.

If the Extractor/Vial Opener jams with the end plug part way out, let the vial warm up before removing the end plug. Forcing out the end plug can damage the Extractor or break the polycarbonate center section. The best way to prevent the Extractor from jamming is to make sure both the Extractor and the threaded end plugs are dry and clean before use.

If the Extractor/Vial Opener jams when being threaded into a cold end plug, it is probably due to water condensing and freezing on the extractor screw. The water freezes when it contacts a cold end plug. If this happens, make a habit of wiping off the screw with a paper towel before each use. Note that the end of the screw is tooled with an "X" cut to help remove any ice that forms on the end plug, but this is not always effective. Chilled vials can also be opened easily and quickly with the optional accessory 6808 Extractor for Large Freezer/Mill Vials.

On rare occasions a flanged end plug will stick in the vial when cold, even if the Extractor screw and end plug threads are dry and clean. If this happens do not force the end plug out, as the vial and/or the Extractor may become damaged. Allow the vial to warm up. The vial can be allowed

6.5 Opening, Emptying, and Cleaning Freezer/Mill Vials (Cont'd)

to warm up gradually, wrapped in several layers of cloth or paper towels. Once the vial is warm, the stuck end plug can be drawn out by hand or with a pair of pliers. When using pliers to extract an end plug, rotate the end plug and pull gently, as if unscrewing a bottle-cap. Forcibly rocking the end plug from side to side can stress the polycarbonate tube and crack it.

CAUTION: Pressure can develop inside a Freezer/Mill vial as it is warming. As pressure builds, an end plug can pop out with force and the sample can be lost. For this reason handle vials with care, and wrap them in paper towel. It is best to open chilled vials immediately after removing them from the Freezer/Mill.

6.5.2 Emptying a Vial

As soon as the flanged end plug is removed from a cold vial, empty the contents of the vial into a suitable container, the quicker the better as condensation on a cold sample occurs rapidly. Often it is helpful to tap the closed end of the vial to release the ground sample. Remember that the impactor may slide out ahead of the contents or with them, and that the longer the sample is exposed to air, the more water it will acquire from condensation. Samples which were tacky, spongy, etc., at room temperature will also return to that state as they warm up, and may agglomerate.

The impactor will have to be separated from the sample and can be handled with gloved fingers, a strong magnet, tongs, or pliers. Tools without a good gripping surface are likely to slip off the impactor. 6870L Accessory Pack included a Magnetic Extractor (6791). This is a rod with a magnetic tip that can be used to remove the impactor from a vial before emptying the contents.

If some condensation on a cold sample is unacceptable, either wait for the sample vial to warm up before opening and emptying it, or open and empty a chilled vial in a glove box filled with dry nitrogen gas.

Small amounts of sample usually adhere to the vial's impactor, end plugs, and plastic cylinder. Brushing or scraping these surfaces of the vial can often recover this fraction of the sample. Again, speed is important to minimize condensation and other effects of warming.

If it is necessary to recover 100% of the sample, let the vial warm up and add some water or other liquid that will not affect the polycarbonate (see Section 6.5.3). Shake the vial, empty it onto some filter paper, and rinse again if necessary to recover the entire sample.

6.5.3 Cleaning the Vials

The Freezer/Mill vials may be superficially cleaned quickly and easily by placing them under running hot water. If the vial is cold a coating of ice will form on the steel parts, but will melt quickly as the water runs.

6.5 Opening, Emptying, and Cleaning Freezer/Mill Vials (Cont'd)

The plastic center cylinders for all vials, and the Poly-Vial end plugs and impactor capsules, are made of polycarbonate. ***While this polymer is very tough at low temperatures, it is sensitive to alcohol, acetone, and other organic solvents, and should be cleaned only with soap and water.*** A mild bleach solution will control organic contamination. Polycarbonate can be autoclaved, but this will weaken it.

Before re-using polycarbonate cylinders, ***always*** inspect them for cracks or other damage. They may last for dozens or hundreds of samples, but as soon as they begin to crack they should be discarded. Liquid nitrogen can enter through cracks and vaporize when vial is removed from Freezer/Mill. As the vial warms vaporized LN will build pressure and an end plug can pop out with force so the sample may be lost.

If sample adheres to the steel end plugs and impactor, they can be cleaned with water and soap or detergent, or with organic solvents. If they must be disinfected or cleaned of any organic residue, they can be washed with bleach or chemical cleaners or autoclaved, but should always be dried immediately after use. The steel parts in the 6751, 6801, and 6881 vials are made from 440C Stainless Steel, a magnetic stainless steel, which is corrosion-resistant but may rust to some extent if left in contact with water for too long. All stainless steel parts should be dried after washing.

The steel parts of the chromium-free 6771, 6871, and 6883 vials can also be washed, disinfected, or autoclaved. **Do Not wash with bleach or chemical cleaners.** Chromium-free steel is not rust-resistant and must always be dried immediately after washing. Store Cr-Free Vial parts in a sealed bag with a desiccant.

Rust on steel Freezer/Mill parts can be removed by scrubbing them with steel wool or an abrasive cleanser. If rusting persists, store the parts in a sealed bag with a desiccant.

6.6 Checking the Liquid Nitrogen Level

The LN should be topped off when needed as a matter of routine. If the programmed grinding cycle includes more than twenty or thirty minutes of actual grinding time, the nitrogen level should be visually checked at about that point and more added if needed. A cumulative grinding time of more than thirty minutes per run is not recommended, as after that much grinding the LN level is close to the point where the LN sensor will shut down the mill. However, if the mill must be run for an extended time, and the LN sensor stops the grinding program, the mill can be refilled with LN and the grinding program resumed where it stopped.

The 6875 Freezer/Mill can be refilled automatically with the 6820 Auto-Fill System, which adds LN to the tub when the LN level is low. The 6820 Auto-Fill System must be installed and calibrated at our factory. A 6875 Freezer/Mill that was not ordered with an Auto-Fill System must be returned to SPEX SamplePrep LLC if installation is desired.

7.0 6820 AUTO-FILL SYSTEM

7.1 Description

The 6820 Auto-Fill System automatically transfers liquid nitrogen (LN) to the 6875 Freezer/Mill during operation, making hand-filling unnecessary. The Auto-Fill mechanism includes a cryogenic valve linked to LN sensors in the tub. When the LN level is low, the valve opens during Precool or cooling stages to let LN into the tub. The Auto-Fill System as supplied also includes a safety valve for the LN transfer hose.

LN transfer hoses can vary with the LN supply tank or line and are therefore not included with the 6820 Auto-Fill System. Four-foot and six-foot transfer hoses of standard design (part numbers 6906 and 6907) can be purchased from SPEX SamplePrep, or custom transfer hoses can be designed and supplied by the user. The LN inlet is a male ½ inch JIC fitting placed low on the right side of the Freezer/Mill, so the outlet of the hose requires a female CGA295 fitting. The 160 liter and 240 liter LN tanks used by many of our customers generally have a male 3/8 inch (9.5 mm) NPT outlet, so SPEX transfer hoses come with a matching female 3/8 inch NPT fitting. Excessively long transfer hoses are not recommended.

LN tanks come in two types, high-pressure and low-pressure. Some LN tanks have valve systems to switch from low pressure to high pressure. ***Always use a low-pressure LN supply, with a delivery pressure of 20-22 psi.***

7.2 Liquid Nitrogen Connection

Install the safety valve on the outlet of the tank or LN line, with the gooseneck tilted up and the valve outlet down (see Figure 14). Then attach the LN transfer hose to the safety valve, and the other end to the inlet on the Freezer/Mill. The safety valve should be higher than the LN inlet on the Freezer/Mill. The purpose of the safety valve is to have an outlet for nitrogen vapor in case the mill and supply tank valves are both shut, and pressure builds up inside the hose.

When installing the safety valve and transfer hose, use Teflon plumber's tape on all joints, and tighten the nuts with a wrench. If the joints leak, tighten them further and/or use more tape.

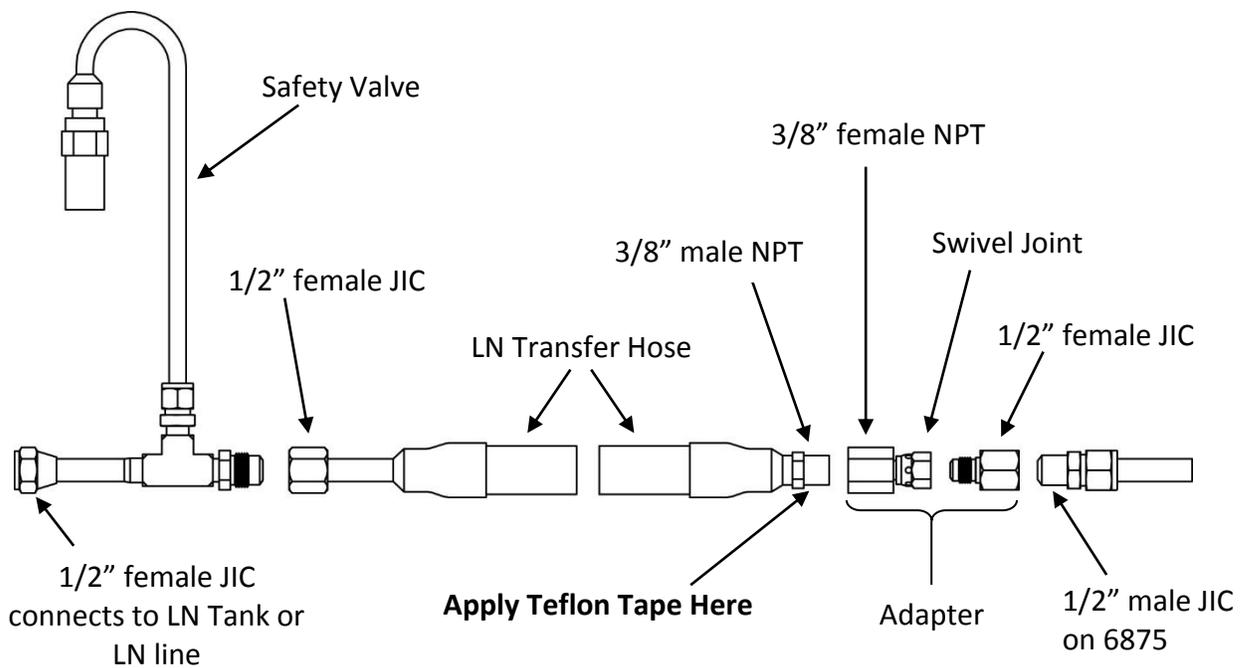


Figure 14 – Liquid Nitrogen Connection (LN Transfer Hose Sold Separately)

8.0 RESOURCES

The Resources screen button is located on the Home screen (Figure 4). To access the Resources screen touch the Resources button. On the Resources screen, as seen in Figure 14, are additional buttons to recall the run history, technical support, accessories (e.g. vials, holders, etc.), and training information (e.g. how-to videos, operating manual). Located at the top right on the Resources screen is the Settings button shaped as a Gear Icon. At the bottom right on the Resources screen is the Home Icon button . Touching the Home Icon returns the user to the Home screen (Figure 4).



Figure 15 – Resources Screen

8.1 Run History

To recall the Run History touch the Run History button. The Date, Time, and Run Protocol data are stored on this screen, as shown in Figure 15. The run information can be exported to other computer devices via the USB Port located on the back of the Controller. To export run data touch the Export History button at the bottom right of the screen. To clear Run History or delete Run History permanently from data storage touch the Clear History button at the bottom left of the screen. To return to the Resources screen touch the back arrow button at the top left corner of the screen.

8.1 Run History (Cont'd)



Date / Time	Pre	Run	Cool	Cyc	Rate	Auto	Dual	Complete
2015-11-28 18:35:45	1	2	1	4	10	false	false	false
2015-11-28 18:36:47	1	2	1	4	10	false	false	false
2015-11-28 18:37:13	4	3	2	5	11	false	false	false
2015-11-29 16:52:19	1	2	1	4	10	false	false	false
2015-11-29 16:52:25	1	2	1	4	10	false	false	false
2015-11-29 16:52:30	1	2	1	4	10	false	false	false
2015-11-29 17:09:23	1	2	1	4	10	false	false	false
2015-11-29 17:09:25	1	2	1	4	10	false	false	false
2015-11-29 17:09:27	1	2	1	4	10	false	false	false
2015-12-01 20:55:55	1	2	1	4	10	false	false	false

Figure 16 – Run History Screen

8.2 Accessories [Electronic Version] – (Available for purchase at additional cost)

To enter the Accessories Screen touch the Accessories button. In the Accessories Screen items are grouped into separate categories (e.g. Grinding Vials, Vial Racks, Extractors), as shown in Figure 17. To open any of the categories listed touch the **+** button to the right of the screen. For example, pressing the **+** button for the Extractors displays the screen for items available in this category. (Figure 18)

To close or minimize the items displayed screen, touch the **-** button top-right of the screen. To return to Resources screen touch the back arrow button located top-left of the screen. (See Section 8 of Manual for additional accessories listing)

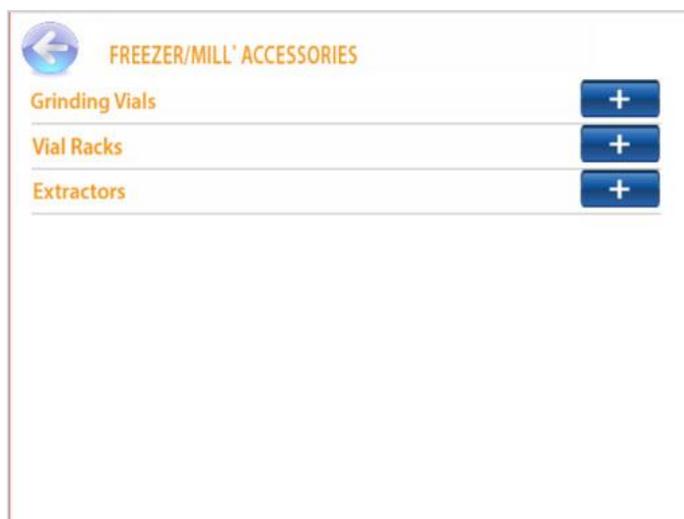


Figure 17 – Accessories screen

8.2 Accessories [Electronic Version] (Cont'd)

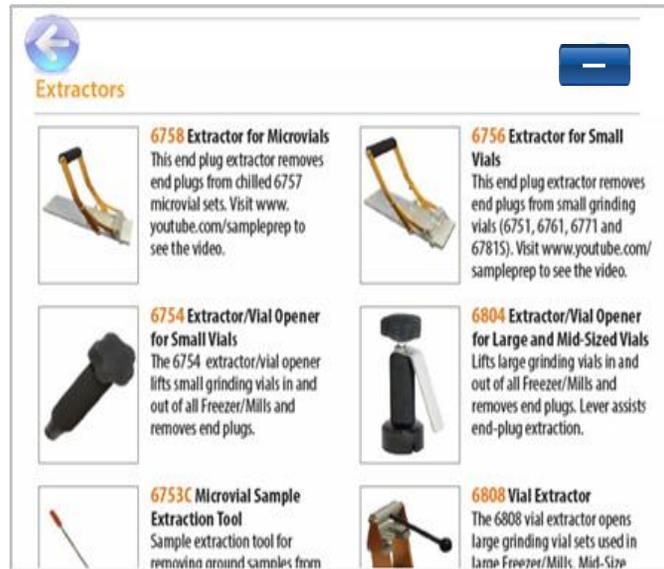


Figure 18 – Extractor Accessories

8.3 Training

To access the Training screen touch the Training button on the Resources screen. In the Training screen the operating manual, instructions and how-to-use videos are listed. To open any of the categories listed on the screen touch the button name, as displayed in Figure 19. To return to Resources screen touch the back arrow button at the top left corner of the screen.



Figure 19 - Training Screen

8.4 System Settings

To access the Settings screen touch the Gear Icon. The System can be upgraded, the time display changed from 12-hour to 24-hour, and Time and Date can be entered or changed, as shown in Figure 20. Touch window to change settings then touch the SET button to lock in the figure. A pop-up window opens confirming, Time or Date resets. To return to Resources Screen touch the back arrow at the top left corner of the screen.

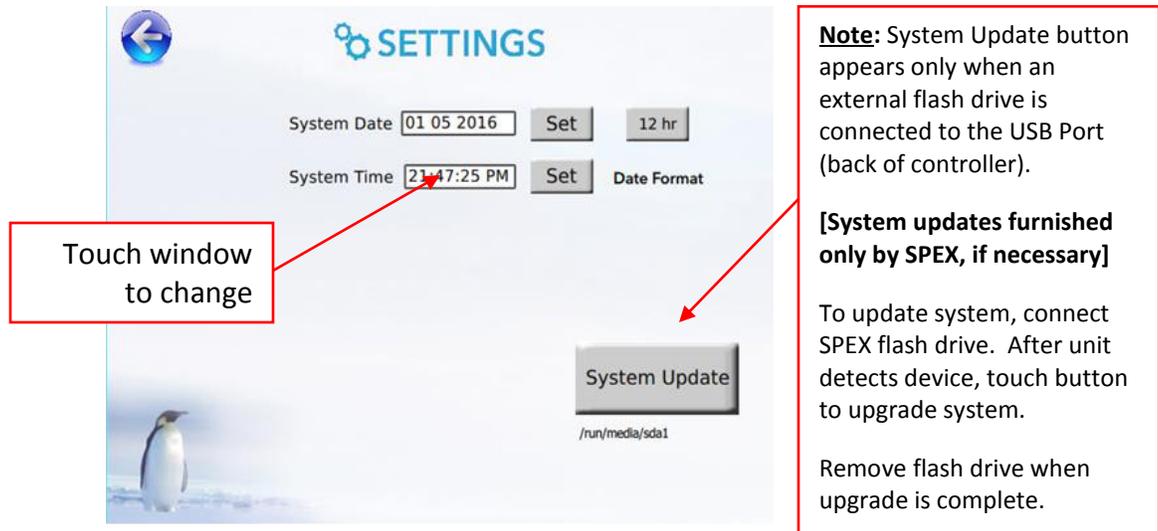


Figure 20 – Settings Screen

8.5 Technical Support

To access the Technical Support screen touch the Tech Support button on the Resources screen. SPEX contact information for the USA Headquarters and Europe Sales Office is listed on this screen (Figure 20). To return to Resources screen touch the back arrow button at the top left of the screen.



Figure 21 - Technical Support Screen

9.0 ACCESSORIES FOR THE 6875 FREEZER/MILL (Available for purchase at additional cost)

A partial list of available accessories is shown here. For a complete listing, please visit our website (www.spexsampleprep.com) or contact us for a copy of the SPEX SamplePrep Handbook of Sample Preparation and Handling.

6757 Microvial Set

Set contains three Microvials arranged in a Vial Holder to allow simultaneous sample processing. Each **6757V** Microvial includes a pair of Stainless Steel End Plugs, one Stainless Steel Impactor, and one Stainless Steel Center Cylinder.

6758 Extractor for Microvials

Used to remove the End Plugs from the Center Cylinders of Small Grinding Vials. Particularly useful for opening cold grinding vials immediately after removal from the Freezer/Mill.

6751 Small Grinding Vial Set

Set contains everything necessary to make one complete Grinding Vial plus three spare Polycarbonate Center Cylinders. Includes **6751P** Stainless Steel Impactor, **6751E** two Stainless Steel End Plugs, and **6751C4** four Polycarbonate Center Cylinders. Sample capacity 0.5–4.0 ml.

6756 Small Extractor/Vial Opener

Used to remove the End Plugs from the Center Cylinders of Small Grinding Vials. Particularly useful for opening cold grinding vials immediately after removal from the Freezer/Mill. Compatible with: **6751** Small Grinding Vial Set, **6761** Small Poly-Vial Set, **6771** Small Cr-Free Grinding Vial Set, and **6871** Small Stainless Steel Grinding Vial. Also compatible with the Vial Holder used in the **6757** Microvial Set.

6755 Vial Rack for Small Grinding Vials

Vial Rack holds up to sixteen Small Grinding Vials for storage and handling. Durable glass-reinforced Acetal construction.

6761 Small Poly-Vial Set

Small Poly-Vial Set contains everything necessary to make one complete Grinding Vial plus three spare Polycarbonate Center Cylinders. Includes one **6761P** Small Polycarbonate Encapsulated Impactor, two **6761E** Small PC End Plugs, and one pack of **6751C4** Small PC Center Cylinders. Sample capacity 0.5 – 4.0 ml.

6771 Small Cr-Free Grinding Vial Set

Small Cr-Free Grinding Vial Set contains everything necessary to make one complete Grinding Vial plus three spare Polycarbonate Center Cylinders. Includes one **6771P** Small Cr-Free Steel Impactor, two **6771E** Small Cr-Free Steel End Plugs, and one pack of **6751C4** Small Polycarbonate Center Cylinders. Sample capacity 0.5 – 4.0 ml.

9.0 ACCESSORIES FOR THE 6875D FREEZER/MILL (Cont'd)

6781S Small Stainless Steel Vial Set

Small Stainless Steel Set contains everything necessary to make one complete Grinding Vial. O-rings on End Plugs seal Vial during grinding. Includes **6751P** Stainless Steel Impactor, Stainless Steel End Plugs, Stainless Steel Center Cylinder, and pack of 10 Silicone O-rings **6781O**.

6801 Large Grinding Vial Set

Contains everything necessary to make one complete Grinding Vial plus three spare Polycarbonate Center Cylinders. Includes one **6801P** Large Stainless Steel Impactor, two **6801E** Large Stainless Steel End Plugs, and one pack of **6801C4** Large Polycarbonate Center Cylinders. Sample capacity up to 50 ml.

6803 Large Stainless Steel Vial Set

Large Stainless Steel Set contains everything necessary to make one complete Grinding Vial. O-rings on End Plugs seal Vial during grinding. Includes **6801P** Stainless Steel Impactor, Stainless Steel End Plugs, Stainless Steel Center Cylinder, and pack of 10 Silicone O-rings **6803s**.

6804 Large Extractor/Vial Opener

Used to remove the end plugs from the Center Cylinders of Large and Mid-Size Grinding Vials. Particularly useful for opening cold Grinding Vials immediately after removal from the Freezer/Mill. Compatible with the following Large Grinding Vials: **6801** Large Grinding Vial Set and **6871** Large Cr-Free Grinding Vial Set. Also compatible with the following Mid-Size Vials when used in conjunction with the **6884** Mid-Size Grinding Vial Adapter: **6881** Mid-Size Grinding Vial Set, **6883** Mid-Size Cr-Free Grinding Vial Set, and **6885** Mid-Size Poly-Vial Set.

6805 Vial Rack for Large Grinding Vials

Vial Rack holds up to six Large Grinding Vials for storage and handling. Durable epoxy-coated steel construction.

6807 Vial Holder

Insert that is placed inside the 6875 grinding chamber to separate and align Small Vials during grinding. Enables multiple sample processing for high-throughput applications. Holds up to four small vials or up to four microvial sets.

6808 Large Extractor/Vial Opener

Used to remove the end plugs from the Center Cylinders of Large and Mid-Size Grinding Vials. Particularly useful for opening cold Grinding Vials immediately after removal from the Freezer/Mill. Compatible with the following Large Grinding Vials: **6801** Large Grinding Vial Set, **6803** Large Steel Vial Set and **6871** Large Cr-Free Grinding Vial Set. The 6808 Extractor is compatible with the following Mid-Size Vials when used in conjunction with the 6808M Kit (sold separately): **6881** Mid-Size Grinding Vial Set, **6883** Mid-Size Cr-Free Grinding Vial Set and **6885** Mid-Size Poly-Vial Set.

9.0 ACCESSORIES FOR THE 6875D FREEZER/MILL (Cont'd)

6870S Small Grinding Vial Accessory Package

Small Grinding Vial Accessory Package contains everything necessary to run Small Grinding Vials in the 6875 Freezer/Mill. Includes one **6754** Small Extractor/Vial Opener, one **6755** Vial Rack for Small Grinding Vials, and one **6806** Multi-Vial Adapter.

6870L Large Grinding Vial Accessory Package

Large Grinding Vial Accessory Package contains everything necessary to run Large Grinding Vials in the 6875 Freezer/Mill. Includes **6804** Large Extractor/Vial Opener and **6805** Vial Rack for Large Grinding Vials.

6870M Mid-Size Grinding Vial Accessory Package

Mid-Size Grinding Vial Accessory Package contains everything necessary to run Mid-Size Grinding Vials in the **6875**. Includes one **6884** Mid-Size Grinding Vial Adapter for Large Extractor/Vial Opener and one **6887** Mid-Size Vial Adapter.

6871 Large Cr-Free Grinding Vial Set

Large Cr-Free Grinding Vial Set contains everything necessary to make one complete Grinding Vial plus three spare Polycarbonate Center Cylinders. Includes one **6871P** Large Cr-Free Steel Impactor, two **6871E** Large Cr-Free Steel End Plugs, and one pack of **6801C4** Large Polycarbonate Center Cylinders. Sample capacity up to 50 ml.

6881 Mid-Size Grinding Vial Set

Mid-Size Grinding Vial Set contains everything necessary to make one complete Grinding Vial plus three spare Polycarbonate Center Cylinders. Includes one **6881P** Mid-Size Stainless Steel Impactor, two **6881E** Mid-Size Stainless Steel End Plugs, and one pack of **6881C4** Mid-Size Polycarbonate Center Cylinders. Requires **6887** Mid-Size Grinding Vial Adapter. Sample capacity 3-25 ml.

6883 Mid-Size Cr-Free Grinding Vial Set

Mid-Size Cr-Free Grinding Vial Set contains everything necessary to make one complete Grinding Vial plus three spare Polycarbonate Center Cylinders. Includes one **6883P** Mid-Size Cr-Free Steel Impactor, two **6883E** Mid-Size Cr-Free Steel End Plugs, and one pack of **6811C4** Mid-Size Polycarbonate Center Cylinders. Requires **6887** Mid-Size Grinding Vial Adapter for grinding in the 6875D and **6884** Mid-Size Grinding Vial Adapter for Large Extractor/Vial Opener.

6884 Mid-Size Grinding Vial Adapter for Large Extractor/Vial Opener

Adapts **6804** Large Extractor/Vial Opener for use with Mid-Size Grinding Vials. Compatible with all Mid-Size Grinding Vials.

9.0 ACCESSORIES FOR THE 6875D FREEZER/MILL (Cont'd)

6885 Mid-Size Poly-Vial Set

Mid-Size Poly-Vial contains everything necessary to make one complete Grinding Vial plus three spare Polycarbonate Center Cylinders. Includes one **6885P** Mid-Size Polycarbonate Encapsulated Impactor, two **6885E** Mid-Size Polycarbonate End Plugs, and one pack of **6811C4** Mid-Size Polycarbonate Center Cylinders. Requires **6888** Mid-Size Grinding Vial Adapter for grinding in the 6875D and **6884** Mid-Size Grinding Vial Adapter for Large Extractor/Vial Opener.

6887 Mid-Size Grinding Vial Adapter

Insert that is placed inside the 6875 grinding chamber to position and align **6881** and **6883** Mid-Size Grinding Vials. Holds One Mid-Size Vial.

6888 Mid-Size Grinding Vial Adapter

Insert that is placed inside the 6875 grinding chamber to position and align **6885** Mid-Size Grinding Vial. Holds One Mid-Size Vial.

6900S Cryogenic Gloves, Size Small

Cryogenic gloves to protect hands and arms from liquid nitrogen exposure. Highly recommended when operating all **SPEX SamplePrep Freezer/Mills®**. Sold in pairs.

6900M Cryogenic Gloves, Size Medium

Cryogenic gloves to protect hands and arms from liquid nitrogen exposure. Highly recommended when operating all **SPEX SamplePrep Freezer/Mills®**. Sold in pairs.

6900L Cryogenic Gloves, Size Large

Cryogenic gloves to protect hands and arms from liquid nitrogen exposure. Highly recommended when operating all **SPEX SamplePrep Freezer/Mills®**. Sold in pairs.

6900XL Cryogenic Gloves, Size Extra Large

Cryogenic gloves to protect hands and arms from liquid nitrogen exposure. Highly recommended when operating all **SPEX SamplePrep Freezer/Mills®**. Sold in pairs.

6906 Short Cryogenic Transfer Hose

4 ft. (1.2 m) long flexible stainless steel hose suitable for transferring liquid nitrogen directly into all **SPEX SamplePrep Freezer/Mills®**. Fitted with a 3/8 in. (9.5 mm) NPT male fitting on one end and a CGA295 female fitting on the other end.

6907 Long Cryogenic Transfer Hose

6 ft. (1.8 m) long flexible stainless steel hose suitable for transferring liquid nitrogen directly into all **SPEX SamplePrep Freezer/Mills®**. Fitted with a 3/8 in. (9.5 mm) NPT male fitting on one end and a CGA295 female fitting on the other end.

10.0 MAINTENANCE

The 6875 Freezer/Mill has been designed to provide trouble-free operation over a long period of time. To assure proper performance it is very important to keep the unit clean. When the Freezer/Mill is at room temperature, any spilled powders or liquids should be wiped up immediately. The internal tub should be wiped clean with a damp cotton cloth after every use.

This should prevent the buildup of any powders, mold/mildew, or other residue over the life of the unit. If any samples, powder materials, or liquids are spilled inside the unit during a sample run, wait until Freezer/Mill is finished for the day, the liquid nitrogen has evaporated, and the mill has warmed up to room temperature before attempting to clean it. Once the unit has attained room temperature, wipe down the unit as indicated previously. After every period of use, the mill will become wet with condensation due to the Freezer/Mill picking up moisture from air when it is cold.

To maintain the exterior of the unit, first disconnect the Freezer/Mill from its electrical source. Then lightly spray with a mild window cleaner or similar product and wipe unit down. If the 6875 Freezer/Mill requires service, please call SPEX SamplePrep Customer Service for assistance.

10.1 Changing the Fuses

If the Freezer/Mill will not operate when the start button is pressed, it is possible that one or both of the fuses may have blown. To access the fuses, first remove the power cord from the back of the Freezer/Mill. Then open the door on the fuse compartment by gently prying it open at the top and flipping it down, as shown in Figure 21. Use a small screwdriver to gently pry the red fuse holder out of the compartment, as shown below. Remove the fuses and check them using a continuity tester. If either fuse is blown or defective, replace both with 3AG 5-amp, 250V slow-blow fuses. Position the fuse holder such that the appropriate operating voltage appears on top. Return the fuse holder to the fuse compartment and close the access door. Check the window of the fuse compartment to make sure that it shows the appropriate voltage. If not, the fuse holder is upside down and must be turned around before attempting to operate the Freezer/Mill.

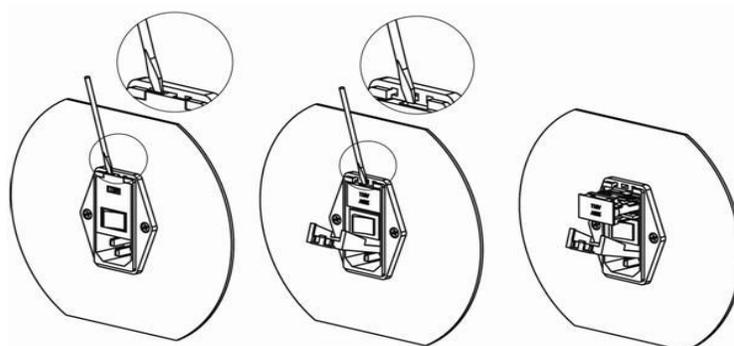


Figure 22 – Fuse Compartment

11.0 TROUBLE-SHOOTING GUIDE

PROBLEM	POSSIBLE CAUSE	POSSIBLE SOLUTION
The unit does not turn on.	No current.	Make sure power cord is plugged into outlet and inlet.
	No current.	Check outlet for power cord and correct as required.
	Power switch not turned on.	Press power switch to ON position.
	Blown fuse.	Replace fuses.
	Blows fuses repeatedly.	Electrical short. Contact SPEX SamplePrep and return for repair.
Screen displays "LID UP".	Safety interlock switch not engaged.	Close the lid and latch.
	Lid is not closed completely.	Push the lid closed and latch into place.
Screen displays "LN LEVEL LOW".	Liquid nitrogen sensor detects inadequate liquid nitrogen level.	Pour more liquid nitrogen into the tub.
Coil gets very warm.	Low liquid nitrogen levels.	Pour more liquid nitrogen into the tub.
Impactor doesn't move back and forth.	Water in vial has frozen impactor.	Remove vial and replace vial and contents with a dry unit or RAP ends of the vial on counter to free impactor.
	Impactor is magnetized.	Remove impactor, turn end-for-end, or demagnetize impactor and re-insert.
	Too much sample is in the vial or sample pieces are too large	Remove some sample from the vial and start again
Flanged End Plug doesn't come off cold vial.	End plug/tube joint too tight when very cold.	Allow vial to warm up.
Blunt end plug doesn't come off room-temperature vial.	Hard to grip	Warm vial under hot water. Use wide-jaw pliers if necessary.
6804 Extractor jams.	Water on extractor screws or bell.	Dry extractor before each use.
	Ice in threaded hole in end plug.	Dry end plug thoroughly before using.
Plastic center cylinder cracks.	Cylinder cleaned with alcohol or other organic solvent.	Use bleach, detergent, and water to clean cylinders.
	Vial assembled when chilled.	Never force end plugs into a cylinder.
	Sample pieces acts as wedge.	Reduce size of sample pieces. Make sure there are no sample particles on cylinder ends.

12.0 QUICK PROTOCOLS

Sample	Sample (g)	Sample size	Freezer/Mill	Vial	Accessory	Grinding Protocol	
Bone	2g	5mm - 8mm	6775	6751 6781S	6756 Extractor	Precool	10 min
						Grinding Cycles	3 cycles (1 min each cycle)
						Cool Time	1 min
						Rate	12 cps
Tooth	1 whole tooth	(as-is)	6775	6751	6756 Extractor	Precool	10 min
						Grinding Cycles	3 cycles (1 min each cycle)
						Cool Time	1 min
						Rate	12 cps
Hair (human, animal)	0.5g	(as-is)	6775	6751	6756 Extractor	Precool	15 min
						Grinding Cycles	3 cycles (2 min each cycle)
						Cool Time	2 min
						Rate	12 cps
Tissue (human/animal)	0.3g	1mm-2mm	6775, 6785, 6785D	6757	6758 Extractor 6807 Holder	Precool	5 min
	5g	10mm	6775, 6785, 6785D	6751	6756 Extractor 6807 Holder	Grinding Cycles	2 cycles (1 min each cycle)
						Cool Time	2 min
						Rate	12 cps
Potatoes (vegetables, or similar foods)	50g	10mm	6875, 6875D	6801 6885	6808 Extractor 6887 Holder	Precool	10 min
	5g	10mm	6775	6751	6756 Extractor	Grinding Cycles	3 cycles (1 min each cycle)
						Cool Time	1 min
						Rate	10 cps
Meat (beef, chicken, fish)	50g	10mm	6875, 6875D	6801 6885	6808 Extractor 6887 Holder	Precool	15 min
	5g	10mm	6775	6751	6756 Extractor	Grinding Cycles	3 cycles (2 min each cycle)
	0.5g	1mm-2mm	6775	6757	6758 Extractor	Cool Time	1 min
						Rate	10 cps
Dog Treats	20g	10mm	6875, 6875D	6801 6885	6808 Extractor 6887 Holder	Precool	15 min
	2g	10mm	6775		6756 Extractor	Grinding Cycles	3 cycles (2 min each cycle)
						Cool Time	1 min
						Rate	10 cps
Plant Leaves	10g	(as-is)	6875, 6875D	6801 6885	6808 Extractor 6887 Holder	Precool	5 min
	1g	(as-is)	6775	6751	6756 Extractor	Grinding Cycles	3 cycles (1 min each cycle)
	0.15g	1mm-2mm	6775	6757	6758 Extractor	Cool Time	1 min
						Rate	10 cps

12.0 QUICK PROTOCOLS (Cont'd)

Sample	Sample (g)	Sample size	Freezer/Mill	Vial	Accessory	Grinding Protocol		
Polyethylene [containers- High Density(HD) and Low Density(LD)]	20g	pellets 3mm - 5mm	6875, 6875D	6801 6881	6808 Extractor 6807 Holder	Precool	15 min	*15min
	2g		*6775	6751	6756 Extractor	Grinding Cycles	6 cycles (2 min each cycle)	*4 cycles (2 min each cycle)
						Cool Time	2 min	*2min
						Rate	10 cps	*12 cps
Thermoplastics (recycleable), example: Plastic Bottles	2.5g	pellets	6775	6751 6781S	6756 Extractor	Precool	15 min	
						Grinding Cycles	4 cycles (2 min each cycle)	
						Cool Time	2 min	
						Rate	12 cps	
Thermoset Plastic (non-recycleable) Example: Epoxy Resins, Phenolic Resins..	0.5g	5mm	6775	6781S	6756 Extractor	Precool	15 min	Typically used in electronic devices
						Grinding Cycles	6 cycles (2 min each cycle)	
						Cool Time	2 min	
						Rate	15 cps	
Plastic Toy/Baby Items	2 to 5	5mm	6775	6751 6781S	6756 Extractor	Precool	10 min	
						Grinding Cycles	4 cycles (2 min each cycle)	
						Cool Time	1 min	
						Rate	12 cps	
Teflon (PTFE)	25g	5mm	6875, 6875D	6801 6881	6808 Extractor	Precool	15 min	15 min
	2.5g		*6775	6751	6756 Extractor	Grinding Cycles	6 cycles (2 min each cycle)	*4 cycles (2 min each cycle)
						Cool Time	2 min	2 min
						Rate	10 cps	*12 cps
Rubber	1g	5mm - 8mm	6775	6751	6756 Extractor	Precool	15 min	
						Grinding Cycles	3 cycles (2 min each cycle)	
						Cool Time	2 min	
						Rate	15 cps	
Carpet	3g	5mm	6775	6751	6756 Extractor	Precool	15 min	
						Grinding Cycles	5 cycles (2 min each cycle)	
						Cool Time	2 min	
						Rate	12 cps	
Wood (chips, pieces)	1.5g	5mm	6775	6751	6756 Extractor	Precool	15 min	
	15g		6875, 6875D		6808 Extractor	Grinding Cycles	3 cycles (2 min each cycle)	
						Cool Time	1 min	
						Rate	12 cps	
Circuit Board	1g	5mm	6775	6771	6756 Extractor	Precool	15 min	
						Grinding Cycles	5 cycles (2 min each cycle)	
						Cool Time	2 min	
						Rate	12 cps	

13.0 WARRANTY

SPEX SamplePrep LLC guarantees its products and new equipment against defects in materials or workmanship for one year from the date of original shipment. Repairs, replacements, or parts are guaranteed for 30 days or for the remaining original warranty period (whichever is greater) for the item that was repaired or replaced. Items not produced by SPEX SamplePrep LLC carry the manufacturer's warranty only. 6875 Freezer/Mill wear parts include the coil.

The customer pays return freight for warranty claims. If the warranty claim is valid, SPEX will pay return freight to the customer. However, SPEX SamplePrep reserves the right to judge whether a malfunction during the warranty period is due to defects in materials or workmanship, or to wear, negligence, or misuse.

13.1 Product Specifications

Every effort has been made to provide complete and accurate product operating information in this manual. However, since specifications are subject to change without notice, changes may be made from time to time to improve the performance, reliability, and function of the product. Therefore, slight changes that are not reflected in the current illustrations should be considered minor and inconsequential for the purposes of this operating manual.

13.2 To Arrange a Return Shipment

We want you to be happy with whatever you purchase from SPEX SamplePrep. Please bring any problem to our attention, but please DO NOT RETURN any item before contacting us for a Return Authorization Number and instructions. Unauthorized returns will be refused. Cost for all return transportation is the responsibility of the customer. Credit for returned merchandise will be issued only after goods have been received and inspected. Returned goods are subject to a 25% restocking charge up to a maximum of \$200.00.

14.0 CONTACT US

Within the United States, write, telephone, or e-mail us at:

SPEX SamplePrep LLC
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Metuchen, New Jersey 08840

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Fax: 732-906-2492

Website: www.spexsampleprep.com

E-mail: sampleprep@spex.com

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Outside the United States and Europe, contact the SPEX SamplePrep representative from whom you purchased your equipment. A list of our current representatives is on our website, www.spexsampleprep.com.