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6850 FREEZER/MILL[®]

OPERATING MANUAL

(Serial Number 03000 and Higher)

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1.0 INTRODUCTION

SPEX CertiPrep Freezer/Mills are cryogenic laboratory mills that cool samples to cryogenic temperatures and pulverize them by magnetically shuttling a steel impactor back and forth against two stationary end plugs. Since the vial is closed, the integrity of its contents is maintained: hazardous or critical samples are easily controlled, clean-up is simplified, and cross-sample contamination is eliminated. Because the vial is immersed in liquid nitrogen throughout the grinding cycle, the sample is kept at cryogenic temperatures and its key aspects preserved. These features have made SPEX CertiPrep Freezer/Mills the most effective in the world, the “mills of last resort” for many normally ungrindable samples, or samples which are altered by the heat generated by conventional grinding.

***NOTE:** Before operating the 6850 Freezer/Mill, familiarize yourself with these instructions. The Freezer/Mill is different in its principles and operation from any other laboratory mill. If you want to proceed directly to grinding, the “Operation” section on pages 18-23 can serve as a general review of the actual operation of the mill.*

We suggest unpacking the mill, then going over its various features with these instructions in hand before attempting to add liquid nitrogen or grind a sample.

Included with the mill is a PVC pipe elbow (1¼”ID) which should be attached to the exhaust vent on the left rear of the mill. We recommend pointing the elbow down and to the left at a 45° angle (see Item 18 in Figure B, page 32). Do not point the elbow up, or it will accumulate moisture that can drain back into the mill.

VERY IMPORTANT: Liquid nitrogen not only embrittles samples through severe chilling, making them “grindable,” but also cools the magnetic coil which powers the 6850 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. This 6850 Freezer/Mill has a liquid nitrogen sensor that keeps the mill from being started when liquid nitrogen falls below a predetermined level. Damage to the coil due to lack of liquid nitrogen is not covered by warranty. We recommend that you have the 6820 Autofill attachment be installed in your unit, if you will be utilizing the unit for extended runs where there will not be anyone to keep track of the LN levels.

SAFETY NOTES:

1) ***Liquid Nitrogen can be hazardous.*** Its boiling point is -195.8° C. When working with liquid nitrogen directly or indirectly, you must wear cryogenic gloves to protect your hands. Be careful not to splash liquid nitrogen on clothes or unprotected skin. Always use cryogenic gloves when handling very cold objects or materials: sample vials, the nitrogen tank valve or hose, chilled Freezer/Mill components, etc. We also recommend safety goggles to protect your eyes.

2) ***Do not attempt to operate the 6850 Freezer/Mill without liquid nitrogen!***

2.0 SPECIFICATIONS

Type of mill: cryogenic impact mill

Grinding mechanism: steel impactor driven by magnetic coils

Coolant: liquid nitrogen

Weight (empty, without vial or coolant): 90 lbs. (40 Kg)

Coil holding capacity: the coil will hold one 6801 Vial or four 6751 Vials.

Grinding Vials:

6801 Vial - actual volume with impactor, approx. 190 ml. Typical sample weights: 20-50 grams for biological samples, 10-20 grams for polymers. The maximum capacity for easily ground samples of moderate density is approx. 100 grams.

6751 Vial - actual volume with impactor, approx. 25 ml. Typical sample weights: 2 grams for biological samples, 1 gram for polymers. The maximum capacity for easily ground samples of moderate density is approx. 8 grams.

Note: in practice, the sample capacity of either vial depends on the properties of the sample, particle-size requirements, and other considerations. As a rule of thumb, in a vial held vertically, the sample should occupy about one third and no more than half the height of the vial.

Typical liquid nitrogen consumption:

15 to 20 liters for initial cool-down and filling of the tub

4 to 6 liters for each hour of operation thereafter

Electrical specifications: **CE Approved**, available in 115 60Hz or 230V 50Hz versions.

Cord: 115V 60HZ version, 3-prong grounded cord supplied

230V 50HZ version, 2-prong European cord supplied

NOTE: Operator is responsible for supplying alternate line cord/plug if required.

Fuses: 115V 60HZ version, 15-amp slow-blow fuse in AC input module

25-amp slow-blow fuse in circuit board in lid.

230V 50HZ version, 8-amp slow-blow fuse in AC input module

25-amp slow-blow fuse in circuit board in lid.

CE Compliance and Certification:

The 6850 Freezer/Mill has been tested for compliance with CE Directives for Industrial, Scientific, and Medical Equipment Emission, and Generic Light Industrial Immunity.

NMi USA, Inc. performed CE testing in 1998 and 2001, and their reports (#ETR370.Spx and #40180-01.Spx) are filed at SPEX CertiPrep, Inc.

Safety Features:

Lid Interlock prevents mill from running if lid is not closed.

Liquid Nitrogen Level Sensor prevents mill from starting when liquid nitrogen level is too low, and shuts the unit off if the tub is emptied during a run.

Off/On switch incorporates a kill switch and circuit breaker

Dimensions: 23 in. (58 cm) x 21 in. (53 cm) x 23 in. (58 cm)

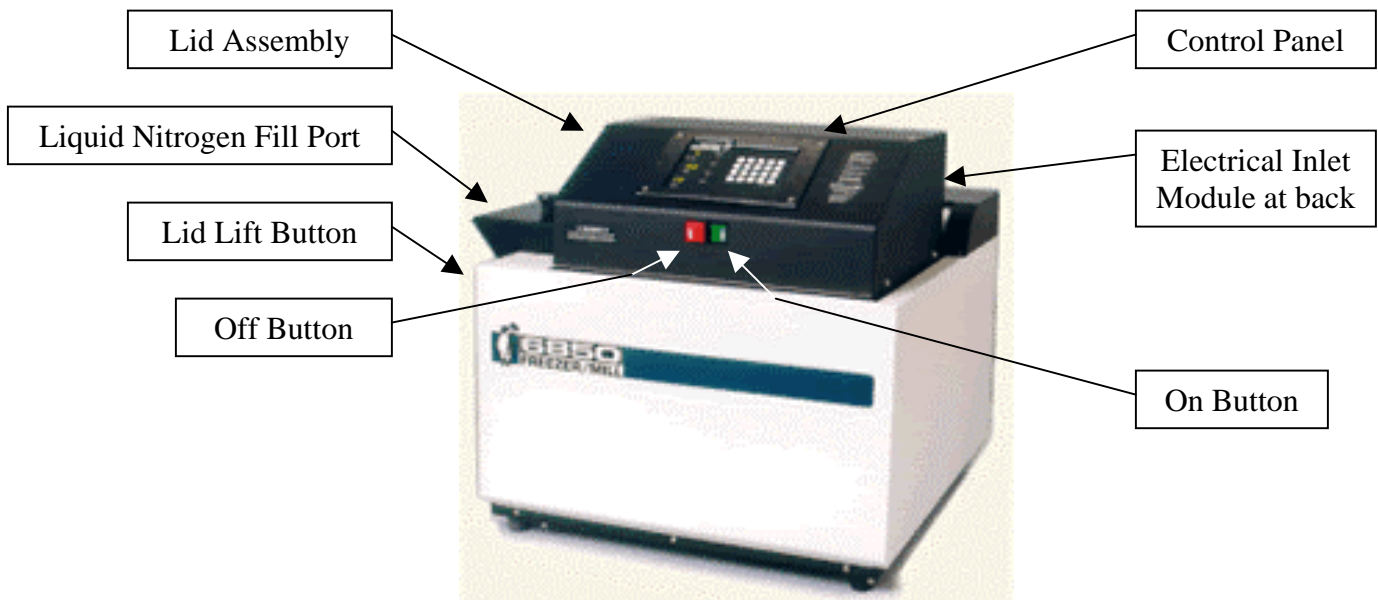
3.0 UNPACKING

Carefully inspect the exterior of the shipping box. Any visible damage to the shipping container should immediately be reported to the carrier. If there is no visible damage, remove all packing documents from the exterior of the box, and after inspection, file in your records.

Open the top of the shipping box. Remove the foamed-in-place packing material by lifting on the retaining plastic sheet. Remove the accessory pack and place nearby. Grasp the mill from both sides, exercising caution while using proper lifting techniques to remove the unit from the carton (the unit weighs 90 lbs). Carefully remove from the shipping box. Save the box for possible future use. Place the mill on the bench top where the mill is likely to stay. Visually check the machine for any hidden damage that may have occurred during shipping. Follow a logical sequence of steps as you inspect the unit (see Figure 1). For example:

1. Inspect the electrical input module for any visible damage.
2. Inspect the top of the cabinet for any visible damage.
3. Are any of the switches damaged?
4. Attach the power cord plug, turn the power on and depress the Lid Up/Down Switch. Does the lid go up and down?
5. Inspect the interior of the tub.
6. Observe and check the coil for any shipping damage.
7. Inspect the accessory pack (see page 6). Compare with the packing list attached to the package. Did you receive what you had ordered?

If everything seems to be in proper order, store the packaging materials in case there is a need to return the unit for future service or repair.



6850 Freezer/Mill

(Figure 1)
SPEX Certiprep

4.0 SETTING UP

4.01 Electrical Hookup and Lid

Plug the mill into a standard 3-prong grounded electrical outlet; we recommend that it be a fused 20-amp circuit for the 115V/60 HZ version. If you have the 230V/50HZ version of the mill, make sure the cord and plug conform to local electrical codes; we furnish the 230V/50HZ 6850 Freezer/Mill with a standard European 2-prong plug with cord.

The 6850 Freezer/Mill weighs about 90 pounds empty. The lid is connected towards the back of the cabinet and opens from the front (see Diagram A, page 31). The electric lid lift is towards the back. The electrical input module, which includes a fuse holder and a connector for the electrical cord is on the back of the cabinet. The fuse is in a tray that can be opened with a fingernail or a small knife blade. Also on the back of the cabinet is the vent from the liquid nitrogen tub.

The top of the cabinet includes the lid, where the ON/OFF switches and the control panel are located. To the left of the lid is a fill port for Liquid Nitrogen. The rocker switch for raising and lowering the lid is also on the left side of the cabinet (see Item 1, Diagram A, page 31), at the top front corner.

4.02 ON/OFF Power Switch and Circuit Breaker

The On/Off switch is located on the front of the lid (see Figure 1, page 5) and consists of a green (On) button and a red (Off) button within a frame. If both buttons are level with the frame, the power is off. To switch the power on, push the green On button in, and the red Off button will pop out. In this exposed position, the Off button can easily be reached for use as a kill switch. To switch the power off, push the Off button in.

The Off/On switch also incorporates an internal circuit breaker. If the circuit breaker trips, the On button will pop back out to the level of the frame while the red Off button stays in the raised position. After diagnosing and correcting the fault, to restore power first push the Off button in, then the On button. You will know the power is on when the LED's display becomes lit.

4.03 Control Panel

The control panel (see Figure 2, page 11) includes (from left to right) alphanumeric LED's displays, indicator LED's, and a keypad. All the programming and operation of the Freezer/Mill are done through the control panel. Programmable parameters include pre-cooling time, grinding time, cooling time between grinding periods, the number of grinding cycles, and the frequency of grinding impacts. The control display also warns if the lid is not fully closed or if there is not enough liquid nitrogen to cool the coil, and can also be used to check the level of coolant in the tub.

4.0 SETTING UP (Cont'd)

4.04 Lid and Lid Control Switch

The lid contains the electronics (with a back-up fuse to protect them) and supports the considerable weight of the magnetic coil. The lid is raised and lowered by an electrical drive located in the rear of the case (see Diagram B, page 32). The Lid Up/Down Switch is the black rocker switch in the top front corner of the left side of the cabinet. To open the lid, rock the switch up and hold it there until the lid is up. To close the lid, rock the switch down and hold it until the lid is down. The lid must be open to load a sample vial into the coil, remove a sample vial, or to fill the tank with liquid nitrogen. If the lid is not fully closed the LED display will show LID and the controls will not function.

4.05 Magnetic Coil, Coil Gate, and Coil Gate Handle

The Magnetic Coil, which is the heart of the Freezer/Mill, is suspended from the lid. On the right-hand side of the coil is a Coil Gate. This gate is operated manually and must be open to insert or remove a sample vial, and closed for the mill to run. The Coil Gate is hinged at the bottom, and secured at the top by an adjustable handle. To open the gate, turn the handle counterclockwise to align it with the slot in the gate, then lower the gate until it rests on the stop below its hinge.

To close the gate, again make sure the handle is aligned with the slot in the gate, and raise the gate past the handle. Now turn the handle ninety degrees, and push the handle down, flat against the gate, to lock it. The gate handle is turned clockwise to tighten the gate, and counterclockwise to loosen it. For the handle to lock properly it should always be positioned at right angles to the slot.

In general it is good practice to lock the gate against the end of the vial to keep the vial end-plugs from pushing out too far out during grinding. Keep the gate snug against the vial but do not over-tighten it. Always lock the gate before running the mill.

During assembly the gate is adjusted so that when a vial is in place the gate will be at right angles to the axis of the vial. If the gate gets out of line it can be adjusted at its anchor point by rotating the entire gate on its mounting bolt, or by loosening the bolt and adding or removing washers.

If you remove a vial partway through a grinding cycle to inspect its progress, the end-plugs may have been pushed out slightly, and you may have to loosen the gate to replace the vial in the coil. If the end-plugs have been pushed out more than about 2 mm, you may want to tap them back in **gently** before placing the vial back in the coil. (Do not hammer the end-plugs in, or over-tighten the gate, as pressure from the locating pins can split the plastic center section of the vial.)

Never touch the coil gate, coil, or sample vial with bare fingers if liquid nitrogen is in use. Always wear gloves when touching any part of the interior of the Freezer/Mill when in use.

4.0 SETTING UP (Cont'd)

4.06 Liquid Nitrogen Level Sensor, and Installation Point for 6820 Autofill System

Inside the tub, at its back on the left, is the Liquid Nitrogen Level Sensor (see item 7, Diagram A, page 31). This is a shielded, perforated tube that senses the level of liquid nitrogen in the tub and relays that information to the controls, where it can be displayed. This detector senses four different levels of liquid nitrogen in the tub. If the coolant level is below Level 3, a grinding program cannot be started, even though this will not stop a grinding program in progress. If the coolant level drops below Level 4, the mill will stop operating even during a grinding program. See “Nitrogen Level Mode” under “Control Modes and Conditions,” page 14.

If your 6850 Freezer/Mill was ordered without the optional 6820 Autofill System, you will notice a flat metal plug on the right side of the rear wall of the tub. This is the installation point for the Autofill filler pipe that brings liquid nitrogen into the tub. The Autofill System is hooked up to a tank of liquid nitrogen and has a valve, activated by the Liquid Nitrogen Level Sensor, which allows more liquid nitrogen into the tub when the level falls. The 6820 Autofill System can be installed at the factory or retrofitted in the field.

4.07 Vial Holder

The Vial Holder is a perforated metal cylinder designed to hold a sample vial in the liquid nitrogen bath so it can cool while another sample is being ground. The Vial Holder hooks over a bracket on the right-hand side of the tub, and is equipped with a handle for lifting it in and out of the liquid nitrogen. The Vial Holder can fit one 6801 vial or four 6751 vials.

4.08 Filler Port

The filler port, to the left of the lid, has a hinged cover (see item 10, page 31). To add liquid nitrogen to the mill without opening the lid, open the cover and pour nitrogen into the port slowly and carefully, to avoid splashing. Normally it is easier to add nitrogen through the open lid, but if (for example) you are in the middle of a long grinding run and need to add liquid nitrogen to the tub, use the filler port. ***NOTE:*** *When you are adding liquid nitrogen through the open lid or filler port, wear gloves, safety glasses, and any protection necessary to avoid spilling nitrogen on your skin or clothes.*

4.09 Nitrogen Vent Elbow

In the filler port or accessory bay, you will find a PVC elbow (Diagram B, item 18, page 32). This fits on the nitrogen vent on the rear of the cabinet and should point down at a 45° angle, as illustrated.

4.10 Vials and Extractor/Rack Accessory Packs

Two different Vials are available for use with the 6850 Freezer/Mill: the standard 6801 Vial, a large-capacity vial which is used one at a time, and the smaller 6751 Vial, which is the standard vial for the 6750 Freezer/Mill but which can be used four at a time in the 6850 Freezer/Mill. Each size of Vial has its own Extractor and Rack, which can be purchased with the mill as part of an Accessory Pack.

4.0 SETTING UP (Cont'd)

If you are using the 6801 Vial you should have the 6800A Accessory Pack; if you are using the 6751 Vial you should have the 6800B Accessory Pack. If you are using both sizes of Vial you can get both Accessory Packs together as the 6800C Accessory Pack. (See pages 27-28)

The standard 6800A Accessory Pack consists of one 6804 Extractor and Vial Opener, and the 6805 Vial Rack which can hold six 6801 Vials.

The 6800B Accessory Pack consists of one 6754 Extractor and Vial Opener, the 6755 Vial Rack which can hold sixteen 6751 Vials, and the 6806 Multi-Vial Adapter to position four 6751 Vials in the 6850 Freezer/Mill's coil.

Individual Vials, Extractors, and other components for either set are available separately.

4.11 6801 Vial and 6800A Accessory Pack

(See page 27)

The 6801 Vial consists of two steel end-plugs, a steel rod-shaped impactor, and a polycarbonate tube. It is usually assembled by inserting one end plug into the tube, placing the impactor and sample in the tube, and inserting the second end plug. The end-plugs are inserted concave side in; the short studs on either side of the plugs control the depth of insertion. The convex side (or in some cases the flat side) of each end-plug has a threaded hole for the screw of the 6804 Extractor and Vial Opener. An assembled 6801 Vial has an internal volume of about 190 ml, and can hold up to 100 grams of some samples.

The 6800A Accessory Pack includes the 6804 Extractor, a single tool with two component parts, the body and the screw. The knob at the narrow end of the body turns the screw at the other end. To pick up an assembled Vial, slip the bell of the Extractor over an end-plug, line up the slots in the Extractor bell with the pins on the end plug, and turn the knob clockwise a turn or two, to thread the screw into the end-plug. The Extractor can now be used to pick up the Vial and (for example) place it in the Vial Holder in the tub for pre-cooling. To release a Vial, turn the Extractor handle counterclockwise. Later model 6804 Extractors have a lever which makes it easier to extract the end-plug.

To open a 6801 Vial, thread the 6804 Extractor's screw into the vial end-plug as described above. Hold the grip of the Extractor with one hand, and with the other turn the Extractor handle clockwise until the end-plug is pulled out of the Vial. Turn the Extractor handle counterclockwise to release the end-plug. If your 6804 Extractor has a lever, give the extractor handle several turns, then squeeze the lever. Repeat until the end-plug is extracted. Caution: as it generally takes both hands to operate the non-levered Extractor, before removing an end-plug you may want to place the Vial in the Sample Rack included with the 6800A. Otherwise the Vial can spill its contents when the end-plug is removed.

Note: whenever you handle a chilled vial, use appropriate protective equipment.

4.0 SETTING UP (Cont'd)

4.12 6751 Vial and 6800B Accessory Pack

(See page 27)

The 6751 Vial, like the 6801 Vial, consists of two steel end plugs and an impactor, with a center cylinder of polycarbonate. It is much smaller than the 6801 and has an internal volume of approx. 25 ml.

The 6800B Accessory Pack consists of a 6754 Extractor and Vial Opener, the 6755 Sample Rack for holding one to sixteen 6751 Vials, and the 6806 Multi-Vial Adapter which is inserted in the 6850 coil to hold one through four 6751 Vials in alignment within the coil. The 6751 Vial and the 6754 Extractor and Vial opener function in the same way as the 6801 Vial and 6804 Extractor and Vial Opener described above in the 6801/6804 section. However, only one of the 6751 end-plugs is threaded for use with the Extractor, so be sure always to place a 6751 Vial in the Freezer/Mill with the threaded end of the Vial out, where the Extractor can engage it. Unlike the 6804 Extractor, the 6754 does not come with a lever.

If you are running 6751 Vials in the 6850 Freezer/Mill, use the 6806 Multi-Vial adapter to keep the vials properly aligned. The X-shaped adapter is simply placed in the coil when needed.

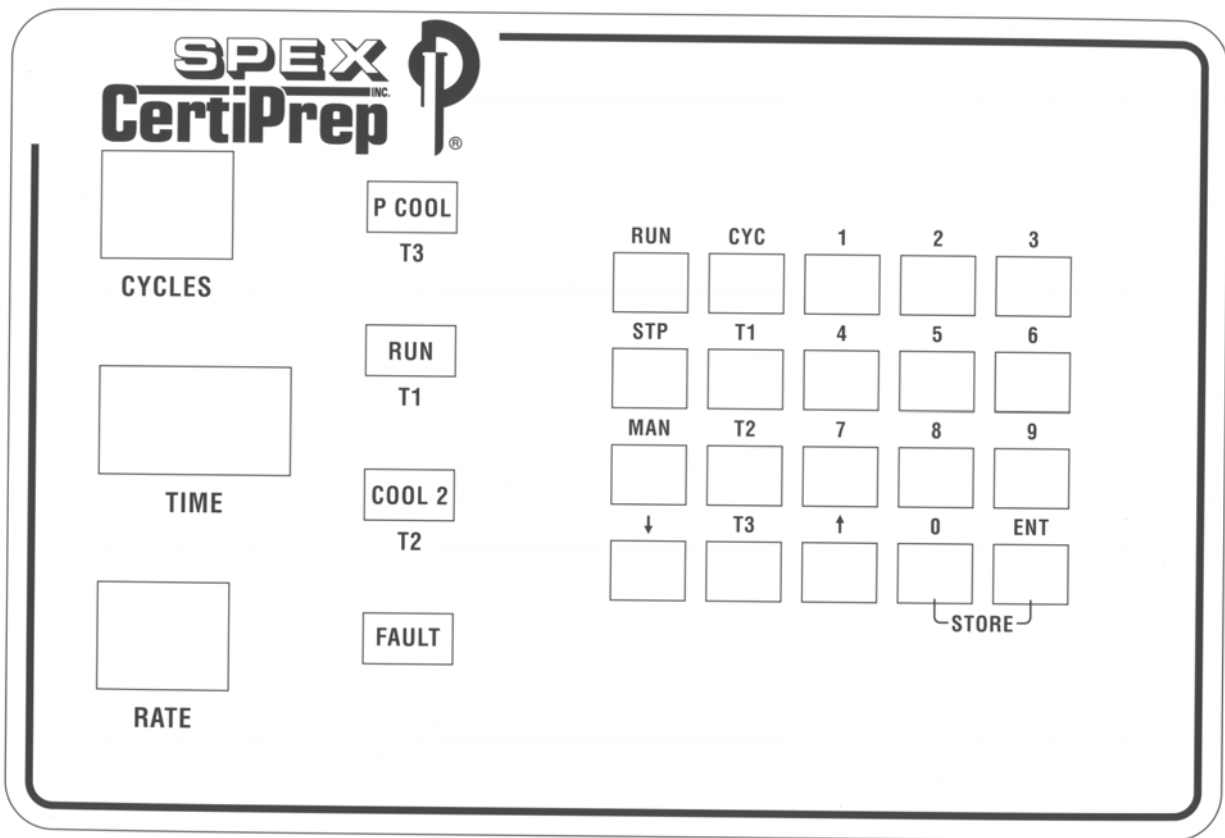
For high-throughput labs we recommend the 6814 Auto-Extractor (page 29). This electrically powered Freezer/Mill accessory takes the place of the manually powered 6754 and 6804 Extractors.

5.0 CONTROLS

5.10 KEYPAD AND DISPLAY

The Key Pad and Display are on the 6850 Freezer/Mill lid, in a rectangular panel with twenty keys, three alphanumeric LED displays, and four indicator LEDs. The keys are used to program and run the Freezer/Mill.

During programming the LEDs display the control settings; when the mill is running the LEDs display the actual conditions of operation. In addition to numbers, these LEDs display five combinations of letters: “LID” (the lid is open); “LN” (liquid nitrogen level); “STO” (store); “END” (end of grinding run); and “PAU/SE” (pause in grinding cycle).



6850 FREEZER/MILL CONTROL PANEL

(Figure 2)

5.0 CONTROLS

5.11 KEYPAD FUNCTIONS BY KEY

Single-Key Functions by Column, Starting From Top Left:

RUN: The RUN key initiates the programmed grinding cycle. This is the **Automatic Mode**. The RUN key also restarts grinding when a **Pause Condition** exists. The mill cannot be started if the lid is not properly closed (**Lid Fault Condition**, p. 15, “LID” is displayed) or the liquid nitrogen level in the tub is too low (see **LN Fault Condition**, p. 14, “LN” is displayed).

STP: The STOP key aborts a grinding cycle and returns the mill to the settings programmed at the beginning of the run. This is the **Stop Mode**. In this mode “END” is displayed for 3 seconds before the display reverts to its programmed settings in **Program Mode**.

MAN: The MANUAL key is a dual-function key which functions as a manual control or pause control. When the mill is at rest in **Program Mode**, pressing the MAN key allows the mill to run for about 3 seconds in **Manual Mode**. When the mill is running in Automatic Mode, pressing the MAN key stops the mill in **Pause Condition** but freezes the settings and allows the mill to be started again where the program stopped by pressing the **RUN** key. PAUSE is displayed during a pause.

↓: DECREASE key. Can be used to decrease programmed values for cycles, grinding time, cooling time, pre-cooling time, and impact frequency.

CYC: The CYCLE key is used to program the number of grinding and cooling periods per grinding program.

T1: TIME 1 key, for programming the time of the grinding period.

T2: TIME 2 key, for programming the time of the cooling period between grinding periods.

T3: TIME 3 key, for programming the time of the pre-cooling period prior to actual grinding.

1 through 0: These NUMERIC keys are used only in **Program Mode**, where in conjunction with the CYC, T1, T2, and T3 keys they can be used to input the program settings. N.B.: the increase (↑) and decrease (↓) keys can also be used to change program settings.

↑: INCREASE key. Can be used to increase programmed values for cycles, grinding time, cooling time, pre-cooling time, and impact frequency.

ENT: Pressing the ENTER key locks current settings in temporary memory.

5.0 CONTROLS

5.11 KEYPAD FUNCTIONS BY KEY (Cont'd)

MULTIPLE KEY FUNCTIONS

0 & ENT: STORE. When the 0 and ENT keys are pressed simultaneously, the current program settings (excluding RATE or impact frequency, which always reverts to 10) are placed in permanent memory, and STO is displayed. Keys 0 and ENT are the two right-hand keys in the bottom row.

Note: any newly entered program setting will displace the prior setting in temporary memory when the ENT key is pushed. Temporary program settings are stored only as long as power to the mill is on. When power is switched off and then on, the program will revert to the settings in permanent memory.

Both temporary and permanent memories have room for only one set of numbers.

2 + 3: LIQUID NITROGEN LEVEL. When the 2 and 3 keys are pressed simultaneously the approximate level of liquid nitrogen in the tub is shown, and LN is displayed. See **Nitrogen Level Mode** below. (Press ENT to return to automatic mode.)

CONTROL MODES and CONDITIONS

Through its keyboard (see page 31 item 6) the 6850 Freezer/Mill can be programmed in **Program Mode**, operated according to program in **Automatic Mode**, interrupted by **Stop Mode**, and tested manually in **Manual Mode**. It also can be shifted into **Nitrogen Level Mode** to display the level of liquid nitrogen in the tub. A **Pause Condition** occurs voluntarily when the operator decides to interrupt (rather than stop) an automatic grinding cycle. A **Lid Fault Condition** occurs automatically when the lid of the mill is not closed. An **LN Fault Condition** occurs automatically when the level of liquid nitrogen drops below the bottom of the coil.

Program Mode: When the Freezer/Mill is first switched on it will display the programmed factory settings for number of cycles, grinding time, cooling time, pre-cooling time, and impact frequency. Cycles, grinding time, and impact frequency appear automatically in the LED display. Cooling time appears when the T2 key is pressed, and pre-cooling time when the T3 key is pressed.

The factory settings are:

| | | | | | |
|-------------------------|----|--------------------|----------|-----------------------|-----------|
| Cycles (CYC) | 3 | Grinding Time (T1) | 2 (min.) | Pre-Cooling Time (T3) | 10 (min.) |
| Impact Frequency (RATE) | 10 | Cooling Time (T2) | 2 (min.) | | |

5.0 CONTROLS

5.11 KEYPAD FUNCTIONS BY KEY (Cont'd)

Any of these five settings may be changed by the user. Changes in control settings go into temporary memory when the ENT key is pressed. They will stay there until the mill is switched off, or can be transferred from temporary into permanent memory by pressing the 0 and ENT keys simultaneously. When the mill is switched on the most recently programmed permanent settings will appear except for Rate, which always reverts to 10. The temporary and permanent memories each have room for just one set of numbers.

Under normal conditions the Freezer/Mill is in Program Mode when switched on, and will remain there until the mill is run, the nitrogen level checked, or a lid default condition occurs. At the end of a programmed run (Automatic Mode) the mill will automatically return to Stop Mode and then Program Mode. If the mill is in any other mode or condition (the display shows "LID" or "LN") it must be returned to Program Mode before it can be started again. If the fault condition is corrected and the display remains in a fault state, press the ENT key.

Automatic Mode: This is the operating mode of the 6850 Freezer/Mill. When the mill is programmed, with the tub filled with liquid nitrogen and a chilled 6801 Vial (or one to four 6751 Vials) in place, press the RUN key. In Automatic Mode the mill will perform the following sequence: pre-cooling, grinding, cooling, grinding, etc., until the programmed number of grinding/cooling cycles is completed. As the mill runs, the display counts down the time left in each period. When the program is complete the LED display will blink END for several seconds, and revert to Program Mode.

Stop Mode: Pressing the STP key while the Freezer/Mill is running in Automatic Mode will stop the run and return the mill to its current programmed settings. The LED display will blink "END" for several seconds, and then the display will return to Program Mode.

Manual Mode: This is a test mode, which allows the mill to operate for about 3 seconds, without beginning a programmed run. With the mill in Program Mode, press the MAN key; the mill will run for several seconds and stop. The chief use of Manual Mode is to verify by ear that the coil is functioning properly, or to set "rate." To do this, place an empty 6801 vial with impactor in the coil; when the mill runs the rattle of the impactor will be clearly audible. The MAN key can also be used to put the mill into Pause Condition when the mill is running.

Nitrogen Level Mode: To determine the amount of liquid nitrogen in the tub without lifting the lid, press keys 2 and 3 simultaneously. In this mode the LED display reads LN and the status lights indicate the level of coolant. If all four lights are lit the tub is full. If the lower three lights are lit, the tub is 2/3 or more full and the liquid nitrogen is above the sample vial. If the lower two lights are lit, the tub is about 1/3 full and the coolant level may be below the sample vial but is still in contact with the bottom of the coil. If the lowest light (the red light marked FLT) is blinking and the LED display shows LN, the liquid nitrogen level is below the level of the coil. This is **LN Fault Condition**, and when the mill is in this state it cannot be started.

5.0 CONTROLS

5.11 KEYPAD FUNCTIONS BY KEY (Cont'd)

Normally if you open the Freezer/Mill's lid to fill the tub with liquid nitrogen, the LED display will show LID. If you want the controls to display the coolant level in the tub with the lid open, close the lid, press keys 2 and 3 simultaneously, and open the lid.

Pause Condition: If you press the MAN key while the mill is running in Automatic Mode, the mill will stop and the LED display will show PAU/SE. To resume the grinding program where it was interrupted, press the RUN key. To end the grinding program and return to the initial program settings, press the STP key. (See Manual Mode, page 14, for the other use of the MAN key.)

During a run you can put the Freezer/Mill into Pause Condition by opening the lid. This can be done to check the degree of grinding, the condition of the vial, whether the impactor is stuck, etc. While the lid is open, the LED display shows LID. When you close the lid, the program conditions at the moment of pause will be displayed except for RUN in the cycles position. Press the RUN button to resume the grinding program.

Lid Fault Condition: When the lid is not completely closed, the LED display will show LID and the controls will not function. This is to prevent the mill from being run with the coil above the level of liquid nitrogen. To return the Freezer/Mill to Program Mode, ready for running, close the lid completely by means of the rocker switch. If the mill remains in Lid Default Condition with the lid fully closed, the interlock switch probably needs adjustment.

LN Fault Condition: When the liquid nitrogen in the tub is below the level of the sample and you press the RUN key, the mill will not start but the FAULT indicator light will blink and the LED display shows LN. This Fault Condition can also be triggered, stopping the mill, when the liquid nitrogen level falls to empty during a run. To correct the Fault Condition you must add liquid nitrogen to the tub, and then press the ENT key to take the keyboard back into operating mode.

6.0 PROGRAMMING

The user may modify any of five parameters: number of cycles (CYC), grinding time per cycle (T1), cooling time per cycle (T2), pre-cooling time (T3), and impact frequency, or rate.

A full grinding program consists of a pre-cooling period followed by several grinding-and-cooling cycles. As the program runs the indicator lights show this as T3, T1, T2, T1, T2, etc.

Cycles and rate (impact frequency) settings are entered as whole numbers. Cooling and pre-cooling times are programmed in tenths of a minute (for example, 1.4 minutes = 84 seconds). These settings are usually entered with the numeric keys; to enter a time of 90 seconds, for example, you would press the 1 and 5 keys. It is also possible to increase or reduce a setting by one digit at a time by means of the increase/decrease (↑/↓) keys. The increase key (↑) cycles 1,2,3, etc., and from 10 to 1. The decrease key (↓) cycles 9,8,7, etc., and from 1 to 10. Pressing the increase (↑) key once would change a setting of 1.5 minutes to 1.6 minutes.

Once a parameter is set, pressing the **ENT** key enters that value in temporary memory, which holds a value until the power to the mill is turned off. To enter a setting in permanent memory, press the **0** and **ENT** keys simultaneously. (Regardless of what is entered in the temporary memory, it is wiped clean every time power is switched off.) When the Freezer/Mill is switched on, the permanent-memory settings are restored. Both the temporary and permanent memories can hold only one setting at a time. The Rate setting always reverts to 10.

CYC: Cycles. One cycle consists of a period of grinding time (T1) followed by a period of cooling time (T2). On the last cycle in a grinding program, the Freezer/Mill skips T2 and goes directly to Stop Mode (the LED display flashes “END” for several seconds) and then into Program Mode. The maximum number of cycles per program is 9. A typical program has 3 or 4 cycles.

To program the number of cycles press the **CYC** key. The cycle’s display will flash. Enter a number using the numeric keys or the increase/decrease arrow keys (↑,↓).

Press the **ENT** key to load the number of cycles into temporary memory, or the and 0 keys simultaneously to load the information into permanent memory.

T1: Grinding Time. This value determines how long the mill will grind during each cycle. It is programmed in tenths of a minute, e.g. 1.5 minutes = 90 seconds. The maximum grinding time per cycle is 10 minutes; a typical grinding time is 2 minutes.

To program Grinding Time press the **T1** key. The TIME display will flash as will the T1 bar LED. Enter a value using the numeric or increase/decrease keys, then press the **ENT** key to load the number in temporary memory.

6.0 PROGRAMMING

(Cont'd)

T2: Re-Cooling Time. This is the length of time between individual grinding periods, programmed in tenths of a minute. The maximum Cooling Time setting is 10.0 minutes. Typical Cooling Time is 1 or 2 minutes, depending on the length of the grinding period and the sensitivity of the sample.

To program Cooling Time press the **T2** key. The Time display will flash as will the T2 bar LED. Enter a value by pressing the numeric or increase/decrease keys (\uparrow/\downarrow), then press the **ENT** key to load the setting in temporary memory.

T3: Pre-cooling Time. This is the length of time before grinding starts, programmed in tenths of a minute. Maximum Pre-Cooling Time is 90 minutes. Typical Pre-Cooling Time settings for most materials are 15.0 minutes for the 6801 Vial and 10.0 minutes for the 6751 Vial. The maximum Pre-Cooling Time is unlikely to be needed.

To program Pre-Cooling Time, press the **T3** key and the Time display will flash as will the T3 bar LED. Enter a value by pressing the numeric or the increase/decrease (\uparrow/\downarrow) keys. Then press the **ENT** key.

Pre-cooling Time is not always needed. Obviously the grinding vial and its enclosed sample must be thoroughly chilled before effective cryogenic grinding can take place. However, the Pre-Cooling step is needed as a regular part of a grinding program only if the vial has not been pre-cooled in some other way. Grinding of a succession of samples can be speeded up by pre-cooling vials in the receptacle inside the liquid nitrogen tub, or even outside the mill in a plastic or metal beaker filled with liquid nitrogen. If you do not need the Pre-Cooling step as part of your grinding program, enter the T3 value as zero.

*Reminder: To enter a program in permanent memory, press the **ENT** and **Q** keys together.*

RATE: Impact Frequency. The number displayed in the LED over RATE is the number of back-and-forth movements of the impactor per second. The factory setting is 10, which is 20 (10 per side) impacts per second. The maximum rate is 15. To change the Impact Frequency, either in Program Mode or while the mill is running in Automatic Mode, press the increase key (arrow-up key) to increase the impact speed, or press the decrease key (arrow down) to slow down the impact speed. The RATE will not be stored as part of a program setting, and will always revert to 10 when the mill is turned off.

7.0 OPERATION

7.1 Trial Run

Once you have mastered the controls of the 6850 Freezer/Mill, we suggest running several test-grinds with samples typical of those you will be grinding. Standard operation of the mill (with factory settings, as shipped) is outlined below.

7.12 Electrical Hookup

Plug the 115V 60HZ mill into a standard 3-prong grounded electrical outlet; we recommend that it be a fused 20-amp circuit. If you have the 230V/50HZ of the mill, make sure the cord and plug conform to local electrical codes; the 230V 6850 Freezer/Mill power cord comes with a standard European 2-prong plug.

7.13 Loading the 6801 Vial

Pick a sample typical of those you will be grinding. For a polymer, measure out 5 to 20 grams, or for biological samples, about twice that much. Assemble a 6801 Vial part way, with one end plug and the impactor, and place the sample in the vial. (Again, the rule of thumb is that a vial held vertically can be filled to about one-third of its height, but no more than half in most cases.) Close the vial with the other end-plug.

Be careful never to introduce any liquid nitrogen, or other gas in liquid or solid form, into the vial when loading it. For example, be very careful about chilling samples in liquid nitrogen before putting them in the vial, or adding “dry ice” (solid CO₂) with which the samples may have been stored. If any such gas is present inside a sealed vial, dangerous pressures can develop when the vial warms up and the gas vaporizes. This can happen during grinding, or after the vial is removed from the mill, and can result in breaking the center cylinder or blowing out an end-plug. Either scenario will disperse the sample, potentially contaminating the mill or your work area. There may also be danger from a forcibly propelled end-plug.

7.14 Off/On Switch, and Factory Settings

Switch on electrical power to the mill by pressing the green START button on the front of the lid down below the level of its frame (see page 3, item 14). Assuming the lid is shut, numbers representing the factory settings will appear in the control display:

CYCLES: 3 (number of successive grinding and cooling periods)

TIME T1: 2.0 (minutes per grinding period)

RATE: 10 (impact frequency in back-and-forth movements of impactor per second)

The other settings are not displayed but are accessible by pressing the indicated keys:

7.0 OPERATION

(Cont'd)

TIME T2: 2.0 (minutes of cooling between grinding periods)

TIME T3: 10.0 (minutes of pre-cooling before grinding starts)

If the LED display reads “LID,” the lid is not completely closed. To close it, press the Lid Up/Down Switch down until the “Lid” message disappears. This switch is located at the top front corner of the left side of the cabinet.

7.15 Inserting the 6801 Vial in the Coil

Open the lid by pressing the Lid Up/Down Switch up, and holding it until the lid is far enough up to give you clear access to the coil. (When the lid is all the way up, it will stop moving and the sound of the motor will change.) Turn the Coil Gate Handle clockwise to line it up with the slot in the Coil Gate, and move the gate past the handle to its resting point. (If the coil is already chilled, use appropriate protective gear.) Slide the 6801 Vial into the coil. (In the case of 6751 Vial sets, insert the 6806 Multi-Vial adapter into the coil before inserting any 6751 Vials.) Now raise the gate past the handle, and snug the gate against the vial by turning the handle clockwise. The gate should be perpendicular to the axis of the grinding vial. Position the handle at right angles to the slot in the gate, and lock the handle by pushing it down against the gate. Don't over-tighten the gate because too much pressure on the end plug pins can crack the plastic center cylinder. The handle is tightened by turning it clockwise, or loosened by turning it counterclockwise.

7.16 Vial Holder

This is the perforated cylinder with handle, which can be hung from a bracket on the right-hand wall of the liquid nitrogen tub. It will hold one 6801 Vial or four 6751 Vials in the liquid nitrogen bath for pre-cooling. If you are intending to run more than one sample, you may wish to load a second 6801 Vial in the Vial Holder at the same time you load your first 6801 Vial into the coil. The Vial Holder has a handle for lifting it in and out of the liquid nitrogen.

7.17 Liquid Nitrogen Coolant Supply

Without the optional 6820 Autofill System installed, liquid nitrogen will have to be poured into the tub by hand. Liquid nitrogen is typically available in either a large tank (e.g. 160 liters) with a flexible steel hose, or in Dewar flasks of various sizes, or both. Either will serve.

The 6850 Freezer/Mill consumes a fair volume of liquid nitrogen: 15 liters or more for initial cooldown, with a maintenance rate of 4 to 6 liters per hour.

There are two ways to add liquid nitrogen to the 6850 Freezer/Mill by hand, either quickly into the tub after opening the lid, or less quickly through the fill port (page 3, item 10). Please remember to wear insulated gloves and other appropriate protective gear when handling liquid nitrogen.

7.0 OPERATION

(Cont'd)

7.18 Filling The Tub With Liquid Nitrogen Coolant

With the lid of the Freezer/Mill open, pour liquid nitrogen into the tub, filling it at first about halfway. Initially the nitrogen will boil off vigorously, but as the tub cools the boiling will subside. Add more nitrogen gradually, to avoid splashing and boiling over. When the tub is filled within an inch of the Pre-Cool Bracket, and boiling is subdued, close the lid slowly by means of the rocker switch until the coil enters the nitrogen. Pause to allow boiling of the nitrogen around the warm coil. When boiling subsides, close the lid the rest of the way. The operating settings (instead of "LID") should appear in the LED display. Some boiling of the nitrogen will still be apparent as a stream of fog being ejected from the vent in the rear of the Freezer/Mill.

When the vapor stream has slacked off (30 seconds or so) check the liquid nitrogen level with the internal sensor by pressing control keys 2 and 3 simultaneously. The LED display is now in Nitrogen Level Mode: it will read "LN" and the indicator lights will show the level of liquid nitrogen in the tub. These levels are: Level 4 (4 lights), full; Level 3 (3 lights), 2/3 full; Level 2 (2 lights), 1/3 full, or enough to cool the coil but not the vial; and Level 1 (1 light), not enough to cool the coil or the vial. *Note that if the nitrogen is below Level 3 a grinding program cannot be started.* If necessary, lift the lid again and add more liquid nitrogen directly, or pour nitrogen through the fill port.

To shift from Nitrogen Level Mode back to Automatic Mode, press the ENT key. You can now run a grinding program.

7.19 Running the 6850 Freezer/Mill

When you press the **RUN** key (with liquid nitrogen in the tub, a loaded vial in the coil, and the factory settings in place), the Freezer/Mill should perform the following steps:

- a) The mill cools for 10 min., while the TIME display counts down the minutes remaining in the pre-cooling period. The T3 indicator light should be on.
- b) The first grinding period begins and lasts for 2 minutes, while the TIME display counts down the time remaining in that grinding period, and the RUN indicator light is lit. The CYCLE display shows 3.
- c) The first cooling period begins and lasts for 2 minutes, while the TIME display counts down and the T2 indicator light is on. The CYCLE display still shows 3.
- d) The second grinding/cooling cycle takes place, the same as in steps b and c combined. The TIME display will count down, and the appropriate RUN and COOL indicator lights will be on in turn, while the CYCLE display shows 2.

7.0 OPERATION

(Cont'd)

e) The third grinding/cooling cycle is like the second (step d) except that the CYCLE display shows 1.

f) At the end of the third grinding cycle, the TIME display will flash END for several seconds, and then the control panel will return to Program Mode, exhibiting the same parameters as when the mill was first started.

During one of the grinding periods you should experiment with the impact frequency by pressing the increase (↑) or decrease (↓) keys to speed up or slow down the speed of grinding. Also while the mill is grinding, try the Pause function by pressing the **MAN** key. The mill should stop and PAUSE appear in the display. Press the **RUN** key to resume grinding, or press the **STOP** key to abort the grinding program.

7.20 Automatic Nitrogen-Level Monitoring During Grinding

Remember that a grinding sequence cannot be started if the liquid nitrogen level is below Level 3. However, during grinding the nitrogen level can drop to Level 2 without interrupting the sequence. If the liquid nitrogen drops below Level 2 during grinding, the Freezer/Mill will stop, the LED will display LN, and the FAULT indicator will flash. Replenish the liquid nitrogen in the tank, close the lid, and press the **ENT** key to return to programming mode and then restart your grinding program.

7.21 Removing, emptying, and cleaning the 6801 or 6751 Vial

At the conclusion of the grinding cycle, signaled by the display flashing END and then returning to its original control parameters, open the lid.

Wearing protective gear, raise the Coil Gate Handle from its lock position, turn it counterclockwise to line up with the slot, and lower the gate. Remove the vial from the coil to the rack, either by hand or by using the Extractor. If you use the Extractor, thread its shaft into the end-plug of the vial, and remove it from the coil. If you have another loaded vial (or vials in the case of the 6751 Vial) ready for grinding, insert it into the coil and close the Coil Gate. Turn the Coil Gate Handle clockwise so the Coil Gate is snug against the Vial, position the handle at right angles to the slot in the gate, and push the handle flat against the gate to lock it. Top off the liquid nitrogen, close the lid, and begin another grinding cycle.

To open the Vial you have just removed from the mill, fit the bell of the 6804 Extractor and Vial Opener over the end of the 6801 Vial (or the 6754 extractor over the end of the 6751 Vial), and line up the grooves in the 6804/6754 Extractor with the pins on the vial's end-plug. Holding the body of the 6804/6754 Extractor with one hand, turn the knob several turns until the screw is engaged. If your Extractor has a lever, squeeze the lever, then turn the knob and repeat until the end-plug is out. If your extractor is without a lever, turn the knob until the end-plug is out. If the screw locks up, don't force it; there is probably ice on the screw or end-plug.

7.0 OPERATION

(Cont'd)

If the ice cannot be chipped away, do not force the Extractor in, but allow the vial to warm up before opening it. An exception to this should be made for samples which may have some gas (nitrogen, CO₂, etc.) trapped in the vial; here it is strongly advised that the end plug be removed when the vial is cold; otherwise an end plug can be blown out, dispersing the sample and creating a projectile hazard.

Here are some suggestions for avoiding "ice-lock." 1) When you have cleaned and dried the end-plugs and are ready to use them again, always make sure there is no water left in the threaded holes. 2) Before using the Extractor, check that the screw and bell are dry. If they are wet, dry them off just before use. The bell and screw of an Extractor get cold when they are used to transfer or open a chilled vial, and moisture will condense on cold metal.

Ensure that the extractor is clean and dry, especially the threaded end, before using.

When the end-plug is removed from the Vial, empty the contents of the vial into a suitable container. You can deal with the impactor by letting it slide out with the contents of the vial, or picking it out of the vial first with a magnet. If the vial and sample are cold the vial should be emptied quickly, as a cold sample will quickly become coated with ice through condensation. (Many samples are not affected by moisture. Others are. Adjust your method accordingly.) Also note that samples which were tacky, squishy, etc., at room temperature before being ground will return to that state as they warm up, and may agglomerate.

Small amounts of sample usually adhere to the vial's impactor, end plugs, and plastic cylinder. Brushing or scraping these components onto a piece of paper or into a plastic bag can often recover this fraction of the sample. In some cases the sample remainder can be washed off and retained on filter paper. Again, if the vial is emptied while cold, speed is important to minimize condensation and other effects of warming.

A cold, emptied vial may be cleaned quickly and easily by placing it under hot running water. A coating of ice will form at first on the steel parts, but the ice will melt quickly as the water runs. If sample adheres to the vial components they can usually be further cleaned with hot water and soap or detergent. Further cleaning and sterilizing may be necessary but as steel and polycarbonate have different physical and chemical properties you may have to clean them differently.

The steel impactor and end plugs can be cleaned like other steel parts, with bleach, disinfectants, organic solvents, etc. They can also be autoclaved. However, once they are cleaned the steel parts should be dried. They are made of 440C stainless steel, a grade that is not completely rustproof. The parts will rust if left wet for any length of time.

7.0 OPERATION

(Cont'd)

6801C and 6751C Center Cylinders are made of polycarbonate. While this polymer is very tough at low temperatures, it is sensitive to strong acids, strong bases, and organic solvents, including alcohol. Some organic solvents will crack polycarbonate and others will dissolve it, so it should be cleaned only with hot water and soap or mild detergent. Polycarbonate can be autoclaved, but may be weakened over time by this procedure; to sterilize polycarbonate we recommend a mild bleach solution. Although these plastic cylinders can last for a long time if properly treated, they should always be inspected just before use, and if they have developed cracks they should be discarded. Cracks in polycarbonate can be seen by holding the cylinders up to the light.

7.22 Optional 6814 Auto-Extractor

The optional 6814 Auto-Extractor, pictured on page 29, will simplify the removal of the end caps for the various vials available for the 6850 Freezer/Mill. The 6814 AUTOEXTRACTOR is a motorized electrical accessory for quick and safe removal of end-plugs from chilled Freezer/Mill vials. The 6814 AUTOEXTRACTOR can open the large 6801 Freezer/Mill vials for the 6800 and 6850 Freezer/Mills, as well as the smaller 6751 vials that are used singly in the 6750 Freezer/Mill and four at a time in the 6800 and 6850 Freezer/Mills. Out of the box, the AUTOEXTRACTOR is set up for 6801 vials, but it includes an easily installed insert for handling 6751 vials.

As with the 6804 and 6754 Extractors, the end plug of a chilled vial is placed in the bell of the extractor, with the locating pins lined up with slots in the bell. Then a rocker switch is pressed up to start the extractor screw, which winches out the end-plug. When the vial is opened, the end-plug can be retrieved by pressing the rocker switch down and reversing the screw. The 6814 Auto-Extractor takes the wrist work out of opening Freezer/Mill vials and is recommended for labs with high sample throughput.

For more detailed operating instructions, please see the Operating Instructions that came with the unit.

If you have any questions about the OPERATION, MAINTENANCE, or SERVICE of your 6850 FREEZER/MILL, please call SPEX CertiPrep at 1-800-522-7739 or 732-549-7144, Extension 465 (Customer Service).

8.0 OPTIONAL 6820 AUTOFILL SYSTEM (OVERVIEW)

8.01 Introduction

For the 6850 Freezer/Mill, the optional 6820 AutoFill System may be purchased factory-installed, or as a retrofit kit for units already in operation. The 6820 automatically fills the Freezer/Mill's tub with liquid nitrogen at startup, and then maintains the fill level while grinding. The AutoFill is linked to the Liquid Nitrogen Sensor in the tub, and when liquid nitrogen gets below a certain level, the sensor opens a valve and admits liquid nitrogen into the tub, then closes the valve when the tub is full. The following overview is based on a 6850 Freezer/Mill with the 6820 Autofill system installed.

8.02 Components

The 6820 AutoFill System, as supplied, consists of two parts: A pipe with a solenoid-operated valve to feed liquid nitrogen into the Freezer/Mill's tub, and a pressure-relief valve for the liquid nitrogen line. **If ordered with your Freezer/Mill, the Autofill System is already factory-installed.** If you purchased the AutoFill Kit separately, **installation instructions are included with the kit.**

As a quick installation overview: The pressure-relief valve must be user-installed in the liquid nitrogen supply line, as described below. The user must also furnish a coupling hose between the liquid nitrogen source and the Freezer/Mill. This hose will need female ½" JIC fittings on both ends. We recommend an insulated flexible hose (available from several companies - we can make recommendations if necessary). The attachment point on the mill for the coupling hose is the male ½" JIC fitting low on the right side of the cabinet. When you are installing the hose we recommend the use of Teflon pipe tape on all threaded joints.

The pressure-relief valve is installed at the opposite end of the coupling hose, between the hose and the nitrogen tank (or fixed nitrogen supply line). Liquid nitrogen tanks usually have a male 3/8" NPT fitting on the outlet, so the pressure-relief valve assembly we supply has a female 3/8" NPT fitting on one end and a female ½" JIC fitting on the other. Install the pressure-relief valve on the tank outlet marked "liquid." (The outlet marked "vent" releases excess nitrogen gas in the tank.) The orientation of the pressure-relief valve is not critical but we recommend that the curve of the gooseneck be the highest part, similar to the form of an inverted "U."

Please note that the pressure-relief valve is a safety feature that must be installed as part of the coupling hose assembly. When the mill and its nitrogen supply are both shut down, some liquid nitrogen remains trapped in the hose. As it warms up it turns to gas, which without a pressure-relief valve could burst the hose. (If you use a pressurized tank to supply liquid nitrogen, it should be a low-pressure tank. Never hook up an Auto-Fill system to a high-pressure tank as it will blow out the SPEX-supplied pressure-release valve.)

Next attach the coupling hose to the pressure-relief valve on one end and the mill on the other. Again, when installing the valve and hose use Teflon tape on all threaded joints, and tighten them with a wrench. If any of the joints leak liquid nitrogen when the mill is in service, tighten them further and/or use more tape.

8.0 OPTIONAL 6820 AUTOFILL SYSTEM

(Continued)

8.03 Operation

Under normal circumstances, operation of the AutoFill System is extremely simple. When the Freezer/Mill is first turned on and **RUN** is pressed, the AutoFill valve will open until the tub is full, **then** the grinding cycle will start. If grinding is programmed in a typical on/off sequence, at any cooling step (T2) the valve will open again if needed. If the mill is used with its factory settings as described on page 10, this is what happens with AutoFill:

- **RUN** pressed.
- LN sensor on. If LN level is low, AutoFill valve opens until tub is full.
- Pre-cool period (T3) begins, and runs for 10 min. (AutoFill keeps level full)
- First grinding cycle (T1) runs for 2 min.
- First cool (T2) runs for 2 min. while AutoFill valve opens if nitrogen level is down.
- Second grinding cycle runs for 2 min.
- Second cool runs for 2 min. while AutoFill valve opens if nitrogen level is down.
- Third grinding cycle runs for 2 min.
- Grinding cycle over. END displayed, then controls revert to original settings.

The first cool-down of the day is a special case. Because the tub and coil are warm, much of the nitrogen will boil off at first and the sensor may produce a false level reading due to the turbulence of the boiling nitrogen. In short, this boiling may cause the valve to shut prematurely, before the tub is as full as it should be. Hence we recommend that for the first run of the day, you always set at least a 10-minute pre-cool (T3) stage, and visually check the liquid nitrogen level most of the way through that first pre-cooling period by raising the lid to see if the tub is full. “Full” means within a few inches of the top of the tub. When you have checked, lower the lid and press RUN to let the pre-cool period run out. If the tub was full when inspected, let the grinding cycle proceed. If the tub was not full, wait the full 10-min. pre-cool period to let the mill stabilize thermally, then switch the mill off, and on again to trigger the LN sensor.

Once this first grinding cycle is over and the mill is thermally stable, the AutoFill System will maintain proper LN levels without supervision.

The 6820 AutoFill System has been designed to work with grinding programs similar to the factory settings, with a pre-cool period and short grinding periods interrupted by short re-cooling periods. If you intend to use the 6850 Freezer/Mill in a way which might bypass the capabilities of the AutoFill System – for example, by having a lengthy pre-cooling period followed by a single long period of continuous grinding - please call us first to make sure your plans and the mill are compatible.

NOTE: For installers of the AutoFill System – the unit comes with its own detailed instruction manual. The manual you are now reading is not intended to provide detailed instruction on the AutoFill, but is intended only to be used as an OVERVIEW. Please follow the instruction manual included with the AutoFill System, as you install the system.

9.0 MAINTENANCE

The 6850 FREEZER/MILL has been designed to provide trouble-free operation over a long period of time. To assure proper performance, perhaps the most important factor is cleanliness. Any spilled powders or liquids should be wiped up immediately. The internal tub should be wiped clean periodically with a damp cotton cloth. The Liquid Nitrogen Fill Port should also be cleaned similarly. This should prevent the buildup of any powders, mold/mildew, or other unsightly gunk over the life of the unit. The complete Coil Assembly should also be wiped clean with a slightly damp cotton cloth from time to time (this is to be done only when the unit is at room temperature!). If you have spilled any sample powder or liquid inside the unit, wait until the unit 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.

To maintain the exterior of the unit, first unplug the FREEZER/MILL, then wipe it down with a mild window cleaner or similar product.

Maintaining the Grinding Vial Sets and the Extractor is basically the normal post use cleanup covered on page 21-23 - Removing, Emptying, and Cleaning the Grinding Vial.

In the rare case that you may need to service the 6850 FREEZER/MILL, please call SPEX CertiPrep Customer Service for assistance with any questions that you may have: 1-732-549-7144, ext. 465.

10.0 Accessories: Vials, Vial Opener and Extractor

6801 Grinding Vial Set

Set includes stainless steel impactor and two end plugs, plus four polycarbonate center cylinders (6801C4). Transparent plastic allows visual check of grinding progress. Sample capacity up to 50 ml.



6801C4 Polycarbonate Center Cylinder

For 6801 vial set: sold in units of 4. It is also available in units of 20 (part number 6801C20). NOTE: Polycarbonate is durable and transparent but it should not be cleaned with organic solvents, including alcohol.

6801 Grinding Vial Set

A complete 6801 Vial consists of two steel end-plugs, a steel rod-shaped impactor, and a polycarbonate tube. The vial is typically assembled by fitting the end-plug onto one side of the tube, inserting the impactor and sample in the tube, and then encapsulating the contents by inserting the other end-plug to the other side. The end-plugs are shaped so that they can be gripped by the 6804 Extractor Vial Opener or the 6814 Auto Extractor.

6800A Accessory Package

Used when operating with 6801 Vial Sets.
Includes 6804 Extractor and one 6805 Vial Rack



6800A Accessory Pack

***Note:** Although the various accessory packages are shown for your information, your unit will arrive only with the accessory package that you have ordered.*

6800B Accessory Package

Used when operating with multiple 6751 vial sets. Includes one 6754 Extractor along with one 6755 Vial Rack and one 6706 Multi-Vial adapter (not shown).



6800B Accessory Pack

10.0 Accessories: Vials, Vial Opener and Extractor

(If not included in your Order, they are Available for Purchase at Additional Cost)

6800C Accessory Package

Optional package at an additional cost. Order when operating with both 6751 and 6801 vial sets. Includes complete 6800A and 6800B Accessory Packages.



6800C Accessory Pack

6751 Grinding Vial Set

Set includes stainless steel impactor and two end plugs, plus four polycarbonate center cylinders (6751C4). Transparent plastic allows visual check of grinding progress. Sample capacity 0.5 ml to 4.0 ml.



6751 Grinding Vial Set

6751C4 Polycarbonate Center Cylinder

For 6751 vial set: sold in units of 4. Also sold in units of 20, 6751C20. Polycarbonate is durable and transparent but should not be cleaned with organic solvents.



6751C4 Polycarbonate Cylinders

6801C4 Polycarbonate Center Cylinders

For 6801 vial set: sold in units of 4. Also sold in units of 20, 6801C20. Polycarbonate is durable and transparent but should not be cleaned with organic solvents.



6801C4 Polycarbonate Cylinders

***Note:** Whenever you handle a chilled vial, we advise using cryogenic gloves.*



6900 Cryogenic Gloves

To Order any of these accessories, call Customer Service at 800-522-7739

10.0 Accessories: Vials, Vial Opener and Extractor

(If not included in your Order, they are Available for Purchase at Additional Cost)

The **6755 Vial Rack** is a glass-reinforced acetal rack. This rack holds up to sixteen 6751 vial sets for storage and handling. One supplied with each 6750 Freezer/Mill, 6800B and 6800C Accessory Package.



The **6814 Auto-Extractor** is a motorized electrical accessory for quick and safe removal of end-plugs from chilled Freezer/Mill vials. The **6814 Auto-Extractor** can open the large 6801 Freezer/Mill vials for the 6800 and 6850 Freezer/Mills, as well as the smaller 6751 vials that are used singly in the 6750 Freezer/Mill and four at a time in the 6800 and 6850 Freezer/Mills.

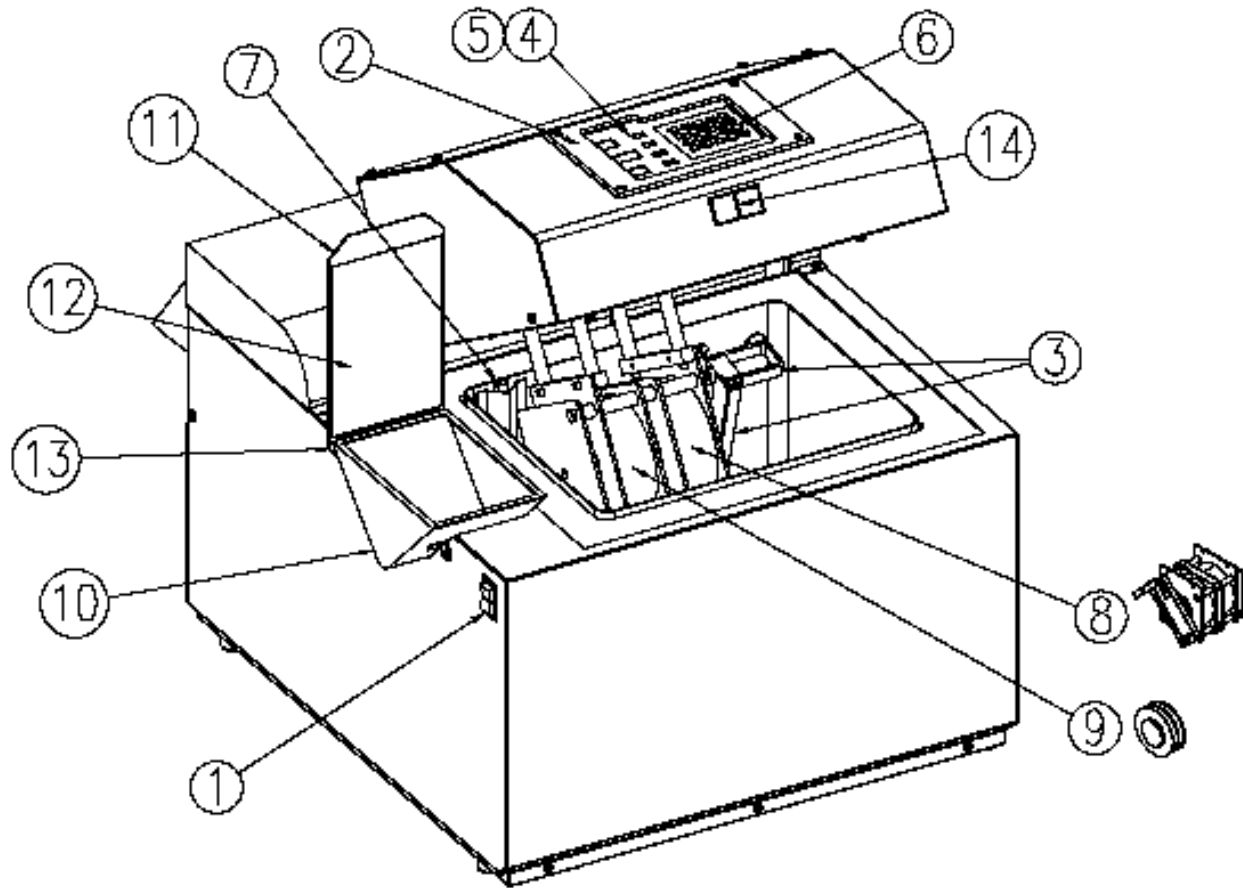


To Order any of these accessories, call Customer Service at 800-522-7739

11.0 Parts List & Item Call-Out Number

| <u>Item</u> | <u>Part No.</u> | <u>Description</u> |
|--------------------|------------------------|---------------------------|
| 1 | 38708 | Lid Switch Assembly 6800 |
| 2 | 38955 | Frame for Timer |
| 3 | 38711 | Gate Assembly 6800 |
| 4 | 38549 | Overlay 6750/6800 |
| 5 | 38562 | Processor Board |
| 6 | 96018 | Keyboard |
| 7 | 38682 | Liquid Level Sensor |
| 8 | 39008 | Solenoid Assembly |
| 9 | 38594 | 6800 Coil |
| 10 | 38944 | Filler Port |
| 11 | 38965 | Lid for Filler Port |
| 12 | 38966 | Magnetic Sheet |
| 13 | 38979 | Hinge on Filler Port |
| 14 | 92706 | On/Off Switch |
| 15 | 38937 | Actuator |
| 16 | 38960 | Vent Pipe |
| 17 | 38961 | Insulation, Vent Pipe |
| 18 | 38962 | Vent Pipe Elbow |
| 19 | 38954 | Flap |
| 20 | 38569 | Driver Board |
| 21 | 93867 | Fuse on Processor Board |
| 22 | 92679 | Switch, Plunger |
| 23 | 38652 | Power Supply |
| 24 | 92941-2 | Relay |
| 25 | 96007 | AC Input (10 Amp) |
| 26 | 96005 | Fuse Holder |
| 27 | 93870 | 15 Amp Fuse (115V) |
| 27 | 93850 | 8 Amp Fuse (230V) |

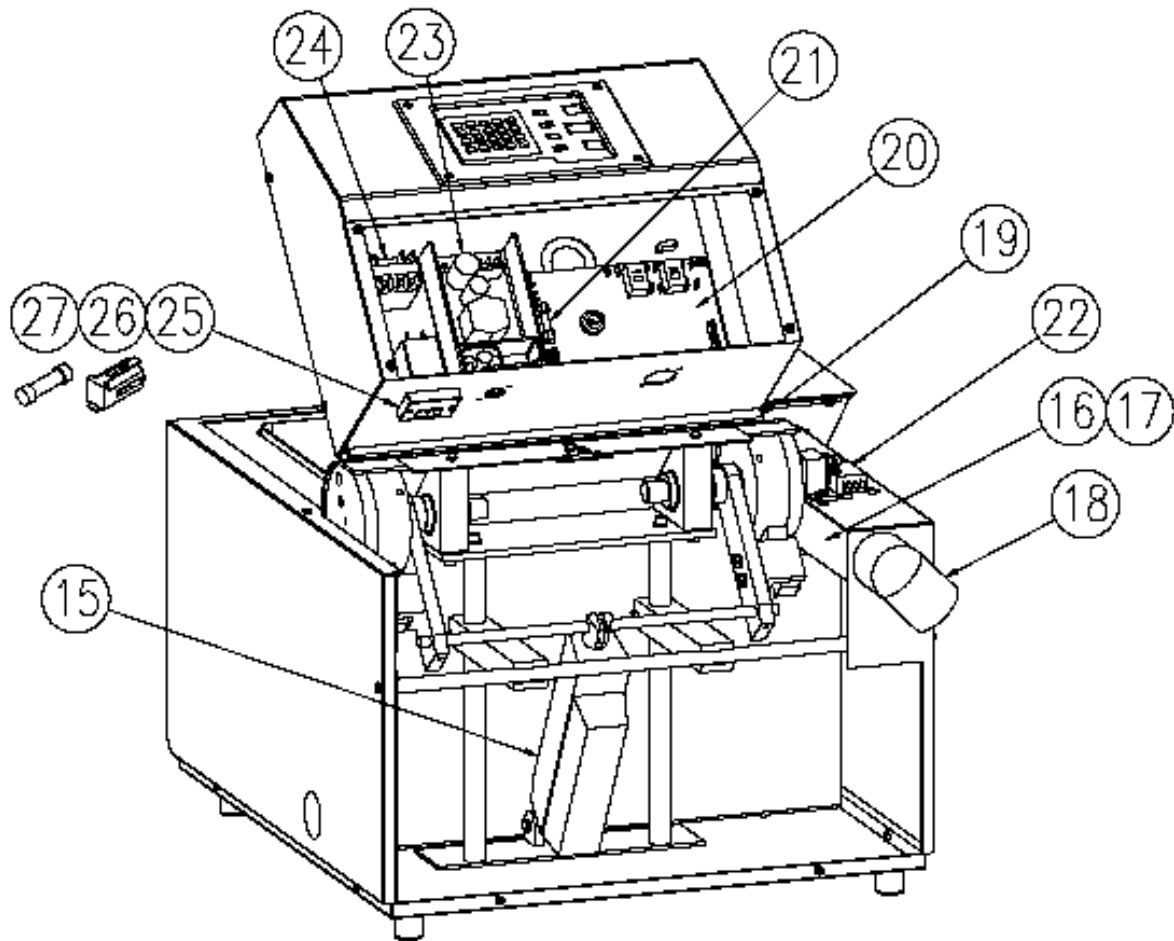
11.01 Diagram A
(Front View)



| <u>Item</u> | <u>Part No.</u> | <u>Description</u> |
|-------------|-----------------|--------------------------|
| 1 | 38708 | Lid Switch Assembly 6800 |
| 2 | 38955 | Frame for Timer |
| 3 | 38711 | Gate Assembly 6800 |
| 4 | 38549 | Overlay 6750/6800 |
| 5 | 38562 | Processor Board |
| 6 | 96018 | Keyboard |
| 7 | 38682 | Liquid Level Sensor |
| 8 | 39008 | Solenoid Assembly |
| 9 | 38594 | 6800 Coil |
| 10 | 38944 | Filler Port |
| 11 | 38965 | Lid for Filler Port |
| 12 | 38966 | Magnetic Sheet |
| 13 | 38979 | Hinge on Filler Port |
| 14 | 92706 | On/Off Switch |

11.02 Diagram B

(Rear view w/covers removed for clarity)



| Item | Part No. | Description |
|-------------|-----------------|-------------------------|
| 15 | 38937 | Actuator |
| 16 | 38960 | Vent Pipe |
| 17 | 38961 | Insulation, Vent Pipe |
| 18 | 38962 | Vent Pipe Elbow |
| 19 | 38954 | Flap |
| 20 | 38569 | Driver Board |
| 21 | 93867 | Fuse on Processor Board |
| 22 | 92679 | Switch, Plunger |
| 23 | 38652 | Power Supply |
| 24 | 92941-2 | Relay |
| 25 | 96007 | AC Input (10 Amp) |
| 26 | 96005 | Fuse Holder |
| 27 | 93870 | 15 Amp Fuse (115V) |
| 27 | 93850 | 8 Amp Fuse (230V) |

12.0 TROUBLESHOOTING GUIDE

| <u>PROBLEM</u> | <u>CAUSE</u> | <u>SOLUTION</u> |
|--|---|--|
| 1 The unit does not turn on | No power | Make sure power cord is plugged into working outlet |
| 2 The unit does not turn on | Blown fuse | Replace fuses |
| 3 The unit does not turn on | Power switch not turned on | Depress power switch |
| 4 The unit does not turn on | Blows Fuses repeatedly | Electrical short (RTF)* |
| 5 The unit turns on but doesn't run | Control Cable is loose or disconnected | Firmly re-attach the Control Cable |
| 6 The unit turns on but doesn't run | Interlock misalignment | Call SPEX service dept. |
| 7 The unit turns on but Impactor doesn't shuttle 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. |
| 8 The unit turns on but Impactor doesn't shuttle back and forth | Too much sample is in the vial or sample pieces are too large | Remove some sample from the vial and start again |
| 9 The unit turns on but Impactor doesn't shuttle back and forth | Impactor is permanently magnetized | Remove impactor, turn end-for-end, or demagnetize impactor and re-insert |
| 10 LED displays "Lid" and "Fault" | Lid is not closed completely | Push the lid closed completely |
| 11 LED displays "LN" | Liquid nitrogen sensor senses inadequate liquid nitrogen levels | Pour more liquid Nitrogen into the tub |
| 12 LED displays "LN" but tub is full of Liquid Nitrogen | Liquid nitrogen sensor senses inadequate liquid nitrogen levels | Faulty LN Sensor operation. Make necessary adjustments. |
| 13 LED displays "LN" and although you have Autofill attachment, tub is empty | Faulty Autofill unit | Call Customer service for repair details |
| 14 Coil is warm | Faulty liquid nitrogen Sensor | Pour more liquid nitrogen into the tub, adjust sensor. |
| 15 End Plug doesn't come off | Water on end plug has frozen unit on | Apply heat with a heater or allow vial to warm up |
| 16 The Lid does not lift up | Water vapor has frozen Lid | Apply some heat to the lid edges until it releases |
| 17 The Lid does not lift up | Fuse is blown | Replace Fuses |

***RTF**=Return to Factory for repair

13.0 WARRANTY

SPEX CertiPrep Inc. guarantees its products and new equipment against defects in materials or workmanship for two years 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 CertiPrep carry the manufacturer's warranty only.

13.01 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 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.

13.02 To Arrange A Return Shipment

We want you to be happy with whatever you purchase from SPEX CertiPrep. 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.

To Contact Us

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Fax: 732-906-2492

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