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6750
FREEZER/MILL®

OPERATING MANUAL

(Serial Number 05000 and Higher)

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1.0 DESCRIPTION



SPEX SamplePrep 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, cleanup 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 SamplePrep Freezer/Mills the most effective in the world. They are the "mills of last resort" for many normally ungrindable samples, or samples whose composition is altered by the heat generated by conventional grinding.

***NOTE**: Before operating the 6750 Freezer/Mill, familiarize yourself with these instructions. The Freezer/Mill is different in its principles and operation from any other laboratory mill. If you're familiar with the mill and want to proceed directly to grinding, the "Running the 6750" section on p. 16 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.

VERY IMPORTANT: Liquid nitrogen not only embrittles samples through severe chilling, making them "grindable," but also cools the magnetic coil which powers the 6750 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 6750 Freezer/Mill has a liquid nitrogen sensor that shuts down the mill when the temperature in the "tub" rises to the point where the coil is at risk. Nevertheless the 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.

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 6750 Freezer/Mill without liquid nitrogen

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2.0 SPECIFICATIONS

Type of mill: cryogenic impact mill

Grinding mechanism: steel impactor driven by dual electromagnets
Coolant: liquid nitrogen
Weight (empty, without vial or coolant): approx. 12 lbs. (5.4 Kg)
Grinding Vials: one 6751, 6751/52, or 6753 vial. (6753 Vial contains 3 microvials.)

Typical Sample Capacity:

6751 - 6751/52 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.
6753 Vial: 100 - 500 mg per micro-vial, depending on sample
Note: actual capacity for a given sample may be smaller or larger, depending on sample properties such as physical form, density, flexibility, etc.

Typical liquid nitrogen consumption:

4-5 liters are required for initial cool-down and filling of the tub.
3-6 liters for each hour of operation.

Electrical specifications: CE Approved, available in 115V 60HZ or 230V 50HZ.

Cord: 115V 60HZ version, 3-prong grounded plug supplied.
230V 50HZ version, 2-prong European plug supplied.

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

Fuses: Two 8-amp slow-blow fuses in AC Input module for 115V,
Two 5-amp slow-blow fuses in AC Input module for 230V,
25-amp slow-blow fuse on circuit board in lid.

Dimensions: 12 in. (30 cm) x 12 ½ in. (32 cm) x 9 in. (23 cm)

CE Compliance and Certification: The 6750 Freezer/Mill has been tested for compliance with CE Directives for Industrial, Scientific, and Medical Equipment Emission, and Generic Light Industrial Immunity.

Safety Features:

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

NOTE: The sensor can shut down the mill during a run, so the 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.

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3.0 UNPACKING

Carefully inspect the exterior of the packing box. Any visible damage should immediately be reported to the carrier. Remove all packing documents from the exterior of the box, and 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 on both sides, and gently remove from the packing box. Place the mill on the benchtop where the mill is to stay. Visually check the machine for any hidden 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 debris or visible damage. Raise and lower the lid to insure that the coil gate (handle with roller) is moving freely and properly.

Each new 6750 Freezer/Mill comes with a 6754 Extractor (page 21) and a 6755 Vial Rack. Grinding vials are necessary for processing samples but must be purchased separately. The 6751 Vial, the 6752 Steel Center Section, and the 6753 Microvial set are described on pages 20-21. The 6814 Auto-Extractor (page 29) is an optional accessory for mechanized removal of end plugs.

Inspect the accessory pack. Remove the Packing List and use it to check that there are no parts missing. If everything seems to be in proper order, then store the packaging materials, in case there is a need to return the unit for future service or repair.



6750 Freezer/Mill

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4.0 SETTING UP

4.01 *Electrical Hookup*

Plug the electrical cord into the inlet on the back of the Freezer/Mill lid, and then into a standard 3-prong grounded electrical outlet. We recommend 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 6750 Freezer/Mill with a standard European 2-prong plug with cord.

The 6750 Freezer/Mill weighs about 12 pounds empty. The lid is hinged (Diagram "B," item 16) on the left side of the cabinet and opens from the right; the lid latch is on the right. The AC Input module, and the power switch (Diagram "A," item 7) are 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 are the vent from the liquid nitrogen tub, and a pneumatic cylinder (Diagram "A," item 13) to control the movement of the lid. The control panel (Diagram "B," item 19) is on top of the lid.

4.02 *Power Switch and Control Panel*

The power switch is the rocker switch on the rear of the Lid. It is marked with two numerical symbols, zero (0) for "off" and one (I) for "on." Press the one (I) side of the switch to turn the power on, or press the zero (0) side of the switch to turn the power off. When power to the mill is on, the control panel display will be lit.

The control panel includes (from left to right) LED displays, and a keypad. The Freezer/Mill is programmed and operated through its control panel, which is described and illustrated in detail on page 9. Programmable parameters include pre-cooling time, grinding time, cooling time between grinding periods, the number of grinding periods, 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 in the tub.

4.03 *Lid and Coil Assembly*

The lid of the Freezer/Mill supports the dual electromagnet assembly, or coil, and coil housing (Diagram "C"). The open space in the coil housing, above the coil, will hold one or two loaded vials, and pre-cool them while another is in the coil, for grinding. Inside the lid are the main electronic board and power supply. To open the lid, push the upper end of the latch (Diagram "B," item 3) away from the lid, then down. If the lower end of the latch is still hooked to the body of the mill, push the lid down and pull the bottom of the latch away from the body of the mill. Then raise the lid, whose movement is damped by a pneumatic cylinder.
The lid must be open in order to load a sample vial into the coil or pre-cooler, to remove a sample vial, or to fill the tank with liquid nitrogen. When the lid is not closed and latched, "LID" will appear in the LED display and blink, the "fault" indicator will blink, and the controls will not function.

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4.03 ***Lid and Coil Assembly***

(Cont'd)

There are two openings in the end of the coil housing. The round opening (Diagram "C" item 30), lined with a phenolic tube, is the center of the magnetic coil; a vial is placed here for grinding. The larger rectangular opening (Diagram "C," item RO) above will hold two vials for pre-cooling (cooling vials prior to grinding).

NOTE: Never touch the coil housing with bare fingers if liquid nitrogen is in use.

The gate (Diagram "C," item CG) holds the vial in place, in the coil, during grinding. When the lid is closed, the gate shuts automatically. When the lid is raised and a vial is in the coil, the coil gate will occasionally stay shut and must be opened manually. (*Always wear gloves when touching a chilled vial or any part of the chilled coil housing!*) To open the gate, pull the gate handle (Diagram "C," item GH) down; it will lever the gate open. The roller on the coil gate handle allows it to retract smoothly into the tub when the lid is being closed.
When you shut the lid, push it down gently, and the coil gate will close. Never force the lid down strongly against resistance, in case the coil gate is jammed.

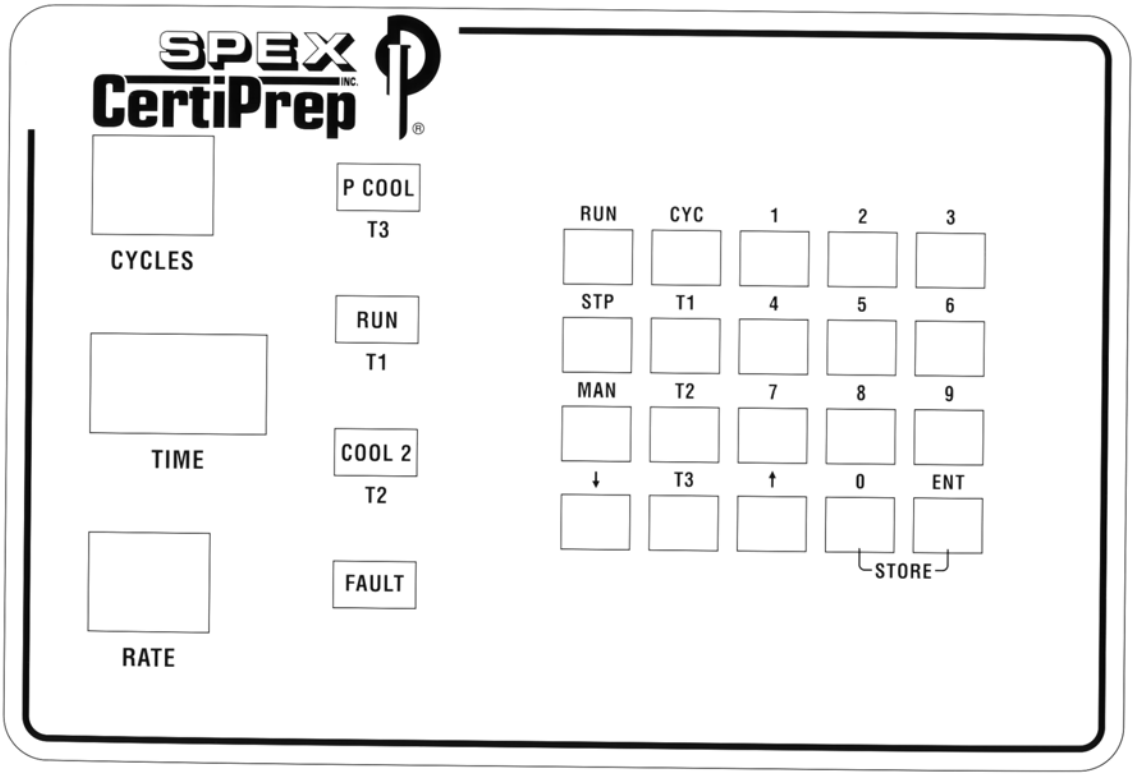
The liquid nitrogen sensor (Diagram "C," item 28) is the small cylinder with wires attached, on the side of the coil housing nearest the hinge. If the liquid nitrogen level in the tub falls to the point where the coil might be damaged from overheating, the control display will flash "LN", the fault indicator will blink, and any grinding cycle in progress will be aborted. To restore the control display, fill the tub with liquid nitrogen to the mark, and press ENT. When RUN is pressed, the grinding program will start over.

NOTE: To keep a run from being interrupted by the liquid nitrogen (LN) sensor, the LN level should be visually checked during extended runs as well as before every run. The 6750 Freezer/Mill will run out of LN after about 20 minutes of actual grinding time, so it is imprudent to run programs with longer grinding time. The Freezer/Mill warranty does not cover damage to the coil caused by operating the mill with little or no liquid nitrogen.

It is possible to test the 6750 Freezer/Mill without liquid nitrogen for a period of several seconds, by putting a 6751 vial in the mill and pressing the MAN key after the LN sensor has been disconnected. Uncouple the pneumatic cylinder from the lid, open the lid all the way, and remove the four screws holding the lid cover. Carefully shut the lid and latch it. Remove the lid cover, and disconnect the LN sensor. This connector is at location P1 on the main board, on the far right corner of the board; its wires are blue and white. Replace the lid cover and press the MAN key; you should hear the clicking of the impactor moving back and forth. Re-attach the connector, lid cover, and pneumatic cylinder before operating the Freezer/Mill with liquid nitrogen. A more detailed description of this procedure is in Appendix 1 on page

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4.10 KEYPAD AND DISPLAY



The keypad and display is a rectangular panel with twenty keys, three numeric LEDs, 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), STO (store), PAU/SE (pause), LN (add liquid nitrogen) and END (the program is finished).

4.11 *KEYPAD FUNCTIONS BY KEY*

Single Key Functions by Columns, 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 ajar (see lid fault condition, page 11) or the liquid nitrogen level is too low (see LN fault condition, page 11).

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.* (Display = "END")

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4.11 ***KEYPAD FUNCTIONS BY KEY***

(Cont'd)

MAN: The MANUAL key, a dual-function key which functions as a pause control when the mill is in automatic mode, or as a manual override when the mill is in stop mode. Pushing this key when the mill is stopped, or the liquid nitrogen level is too low for regular operation, activates the coil for about three seconds. This brief running period can be used to test the coil: place an empty vial with impactor in the coil, and if the coil is functioning, the typical rattling noise will be heard for several seconds when the MAN button is pressed.

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

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

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

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

T3: TIME 3 key, for programming 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. The INCREASE (↑) and DECREASE (↓) buttons can also be used to increase or decrease 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.

MULTIPLE-KEY FUNCTIONS

ENT + 0: STORE. When these keys are pressed simultaneously, the current program settings are placed in permanent memory. (Display = "STO")

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.

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MULTIPLE-KEY FUNCTIONS
(Cont'd)

While learning to use the keypad, remember that if you press the RUN key to begin a grinding sequence when there is no liquid nitrogen in the mill, the liquid nitrogen sensor will shut down the controls, the fault indicator will blink, and the display will flash "LN" for Liquid Nitrogen. To restore the normal control display, press ENT (enter). To operate the mill, place a vial in the coil, fill the tub to the mark with liquid nitrogen, and press RUN. Note that with no vial in the coil, the Freezer/Mill makes no noise when the coil is energized. The only indication it is running will be in the LED displays.

DO NOT RUN THE UNIT WITHOUT THE VIAL AND IMPACTOR, OTHERWISE THE COILS MIGHT BE DAMAGED.

CONTROL MODES and CONDITIONS

Through its keyboard the 6750 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. 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 properly shut. A liquid nitrogen fault condition occurs when there is no liquid nitrogen in the tub, or not enough liquid nitrogen to chill the coil properly.

Program Mode: When the Freezer/Mill is switched on for the first time it will display (and operate according to) the preprogrammed factory settings. These settings are:

Cycles (CYC)	3	Grinding Time (T1)	2 (min.)	Pre-Cooling Time (T3)	15 (min.)
Impact Frequency (RATE)	10	Cooling Time (T2)	1 (min.)		

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 user, following the instructions for programming, may change any of these five settings. 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 ENT and 0 keys simultaneously. When the mill is switched on, the most recently stored permanent settings will recur, with the exception of impact frequency that always reverts to 10.

Under normal conditions the Freezer/Mill is in Program Mode when switched on, and will remain there until the mill is run, the liquid nitrogen level drops enough to trigger the LN sensor, 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"), the fault must be corrected and the mill 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 ENT.

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CONTROL MODES and CONDITIONS

(Cont'd)

Automatic Mode: This is the operating mode of the 6750 Freezer/Mill. When the mill is programmed, the tub is filled with liquid nitrogen, and a chilled grinding vial is in place, press the RUN key. In Automatic Mode the mill will perform the following sequence: pre-cooling, grinding period 1, cooling, grinding period 2, etc., until the programmed number of grinding/cooling cycles is completed. The LED display will then blink "END" for about five 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 about five 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, whether the mill is filled with liquid nitrogen or not. 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. To do this, place an empty 6751 vial with its impactor in the coil; when the mill runs, the rattle of the impactor will be clearly audible. See Pause Condition (below) for an alternate use of the MAN key.

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 read "PAUSE." 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 Control, above, for the other use of the MAN key.)

Lid Fault Condition: When the lid is lifted, or if it is down but the safety interlock is not engaged, the LED display will read "LID" and the mill cannot be run. This is to prevent the mill from being accidentally run with the coil above the liquid nitrogen, or with a vial not held in place by the coil gate. To return the Freezer/Mill to Program Mode, ready for running, close and latch the lid. If the mill remains in Lid Fault Condition with the lid latched, the lid interlock switch may be out of adjustment. If the lid opens unexpectedly during a run, the mill will stop. To resume the run where it was interrupted, close and latch the lid; "RUN" will appear in the display. Press the RUN key to resume grinding.

Liquid Nitrogen Fault Condition: If there is no liquid nitrogen in the Freezer/Mill, or the level of liquid nitrogen in the tub is too low to cool the coil and vial properly, the display will blink "LN" when the RUN key is pressed. To resume operation, fill the tub to the mark with liquid nitrogen, close and lock the lid, and press the ENT key.

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NOTE: Because the LN sensor will stop and abort a run if the level of liquid nitrogen level falls too low, it is important to top off the liquid nitrogen before every run, and at regular intervals during extended runs. In no case should a program include more than twenty minutes of total grinding time (for example, 10 cycles at 2 minutes each in T1).

4.12 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. To program cycles, T1, T2, or T3 push the function key and then the numerical keys. To change impact frequency, just press the INCREASE (↑) or DECREASE (↓) key.

Note: A full grinding program consists of a pre-cooling period followed by several grinding cycles; each cycle consists of a grinding period followed by a period of cooling, except at the end of a program where no further cooling is needed.

Cycles and 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 = 1 minute and 24 seconds. These settings may be entered directly with the numeric keys, or adjusted with the INCREASE/DECREASE (↑↓) keys. In programming cycles or frequency the INCREASE key (↑) advances 1,2,3, etc., up to 15, and back to 1, while the DECREASE key (↓) cycles 9,8,7, etc., and from 1 to 15. If you try to enter a setting outside the programmable range the mill will "beep."

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 transfer a setting from temporary to 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, with the exception of impact frequency, which is always stored as 10. Both the temporary and permanent memories can hold only one setting per parameter.

Any programmed change in cycles, grinding time, cooling time, or pre-cooling time must be individually placed in temporary memory as it is entered by pressing the ENT key. All such programmed changes can be cumulatively transferred to permanent memory by pressing the ENT and 0 keys simultaneously after all changes have been made.

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 Program Mode. The maximum number of cycles per program is 90. A typical program has 3 or 4 cycles. To program the number of cycles press the CYC key. The cycle 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. (If you want to store this information in permanent memory right away, press the ENT and 0 keys simultaneously.)

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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 per cycle.

4.12 PROGRAMMING

(Cont'd)

To program the grinding time, press the T1 key; the TIME display will flash as will the T1 LED. Enter a value using the numeric or INCREASE/DECREASE keys (↑↓). Then press the ENT key to load the number in temporary memory. (If you immediately want to enter this setting in permanent memory, press the ENT and 0 keys simultaneously.)

T2: Cooling Time. This is the length of time between individual grinding periods, and is programmed in tenths of a minute. The maximum cooling time setting is 10 minutes. Typical cooling time is 1 or 2 minutes or 1/3 to ½ of grinding time T1, depending on the length of the grinding period and the sensitivity of the sample to slight temperature increases.
To program cooling time press the T2 key; the TIME display will flash as will the T2 LED. Enter a value by pressing the numeric or INCREASE/DECREASE keys (↑↓). Now press the ENT key to load the setting in temporary memory. (If you want to store this setting in permanent memory immediately, press the ENT and 0 keys simultaneously.

T3: Pre-Cooling Time. This is the length of time before grinding starts, programmed in tenths of a minute. Maximum pre-cooling time is 30 minutes. Typical pre-cooling time for biological samples is 10 minutes, and for polymers is 15 minutes. Certain silicones and very flexible polymers may require more pre-cooling time.

To program pre-cooling time press the T3 key; the TIME display will flash as will the T3 bar LED. Enter a value by pressing the numeric or the INCREASE/DECREASE (↑↓) keys. Now press the ENT key to enter the value in temporary memory. If you want to store this setting in permanent memory, press the ENT and 0 keys simultaneously.

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 pre-cooling slot in the coil housing, or even outside the mill in a plastic or metal container filled with liquid nitrogen. If you don't need the pre-cooling step, enter T3 as zero.

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 actual impacts per second (one to each side). To change the impact frequency, either in Program Mode or while the mill is running in Automatic Mode, press the INCREASE key (↑) to speed it up, or the DECREASE key (↓) to slow it down. The maximum setting is 15. The impact frequency rate is not stored in permanent memory, but always reverts to the factory setting of 10. (Whenever the 6750 Freezer/Mill is switched off and then on, the program in permanent memory will include the set values for cycles, grinding time, cooling time, and pre-cooling time, but the impact frequency will always come up as 10.)

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5.0 OPERATION

5.01 *Trial Run*

Once you've mastered the controls of the 6750 Freezer/Mill, we suggest several test-grinds with samples typical of those you will be grinding. The general operating procedure is outlined below.

5.02 *Electrical Hookup*

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 6750 Freezer/Mill with a standard European 2-prong plug with cord.

5.03 Adding Liquid Nitrogen Coolant.

Liquid nitrogen is likely to be available to you either in a large tank (e.g. 160 or 240 liters) with a flexible steel hose, or in 20-liter or 30-liter Dewar flasks, or both. Either will serve. The 6750 Freezer/Mill consumes a moderate volume of liquid nitrogen: 4 to 5 liters for initial cool-down, with a maintenance rate of 2 to 4 liters per hour, depending on use. We recommend an adequate supply.

Liquid nitrogen is added to the 6750 Freezer/Mill with the lid open. There is a fill mark about an inch below the lip of the tub. If the liquid nitrogen is much above this mark when the lid is closed, the nitrogen is likely to splash outside the tub. *Always close the lid slowly, to avoid splashing.*

5.04 *Filling the Tub with Liquid Nitrogen*

Unlock the latch by pushing the thumb tab at its top away from the cabinet, and then release the lid by pulling the bottom of the latch away from the cabinet. If the bottom of the latch is still engaged, push the lid down to free it. Lift the lid slowly; a pneumatic cylinder controls its movement. The coil mechanism (Diagram "C") is attached to the lid and will move upward with it. If there is no vial in the coil, the coil gate will probably drop open when the lid is lifted. If the coil gate remains closed, pull the coil gate handle outward to lever the gate open.

When the Freezer/Mill tub is empty, pour liquid nitrogen into the tub about a third of the way. 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 nitrogen is up to the fill mark and boiling is subdued, push the lid down gently, stopping if splashing becomes excessive. When the lid is closed and latched, further nitrogen boiling will be visible as a stream of condensed water vapor ("fog") rushing from the vent in the rear of the Freezer/Mill; this is normal. When the vapor stream has subsided, lift the lid again and top off the liquid nitrogen in the tub, filling it to the mark on the inside wall of the tub.

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5.0 OPERATION

5.04 Filling the Tub with Liquid Nitrogen (Cont'd)

To assist in pre-cooling the vials, you might wish to place a vial in the coil, and two others in the pre-cooling slot (Diagram "C," item RO), before filling the tub with liquid nitrogen.

5.05 *Loading the Vial*

Pick a sample typical of those you will be grinding. For a polymer, measure out 1 to 2 grams, or for bone and tissue samples (plant or animal), perhaps twice that much. Keep in mind that in practice, the various parameters (sample size, pre-cooling time, grinding times and cycles, etc.) are customized to match particular samples. Typically, the correct amount of sample will take about 1/3 of the vial volume (with impactor in vial).

If the sample can be handled at room temperature, assemble the center cylinder and blunt end-plug of a 6751 Vial (see Page 4). Add the sample and impactor to the vial, and close it with the flanged end-plug. Shake the vial to make sure the impactor has room to move back and forth. If the sample is cold, and must be kept cold throughout the grinding process, the procedure is more elaborate. Assemble the center cylinder and blunt end-plug, as above, then chill the end-plug and about an inch of the cylinder in a shallow liquid nitrogen bath. At the same time, chill an impactor. The vial rack that comes with the 6750 Freezer/Mill can be placed in an insulated container and used to hold the vials upright during chilling. As soon as the submerged end of the vial is chilled, drop in the cold sample and 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 you are careful not to let the sample touch the warm end plug before it has cooled down. *Very important: never force an end plug (warm or cold) into a cold polycarbonate cylinder. Polycarbonate shrinks when chilled, and can crack if stressed. When you follow this procedure for loading cold samples, if the cylinder has cooled to the point where the end-plug fit is too tight, warm the end of the cylinder before inserting the end-plug.*

A variant of the above technique 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.

Before loading the vial in the mill, put on cryogenic gloves. Unlatch and lift the lid of the mill, and open the coil gate. Now pick up the vial with your gloved hand or the Extractor (page 17), and insert the vial in the coil. If you have additional vials you wish to pre-cool, pick them up the same way and insert them in the pre-cooling slot in the coil housing. Check the liquid nitrogen level and top it off if necessary. Gently press the Freezer/Mill lid closed; the coil gate will shut automatically, and the coil gate handle will retract into the tub. If the lid resists closing, make sure the vial is inserted all the way in the coil. You are now ready to begin a programmed grinding cycle.

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5.0 OPERATION

5.06 *Off/On switch, and factory settings*

If you have not done so, switch on electrical power to the mill by pressing the rocker switch in the rear of cabinet. Numbers representing the factory settings should appear in the control display.

Visible on the LED display:

Cycles: 3 (number of successive grinding periods)
Time 1: 2.0 (minutes per grinding period)
Rate: 10 (impact frequency in cycles per second)

Accessible through keypad by pressing indicated keys:

T2: 2.0 (minutes per cooling period between T1 grinding periods)
T3: 15.0 (minutes of cooling before grinding starts)

Adjust the settings as desired before operating the mill.

5.07 *Running the 6750 Freezer/Mill*

Assuming there is liquid nitrogen in the tub and a loaded vial in the coil, and the factory control settings are retained, the Freezer/Mill should perform the following steps when the RUN button is pushed:

a) The mill cools for 15 min., while the TIME display counts down the time 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 indicates 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 indicates 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 T2 indicator lights will be on in turn, while the CYCLE display reads 2.
e) The third grinding/cooling cycle is like the second (i.e. Step d) except that the CYCLES display reads 1.
f) At the end of the third cycle, the mill will emit four beeps and the TIME display will flash "END" for several seconds. Then the control panel will return to Program Mode, exhibiting the same parameters as when the Freezer/Mill was switched on.

During one of the grinding periods you should experiment with the impact frequency by pressing the INCREASE (↑) and/or DECREASE (↓) keys to speed up or slow down the speed of grinding.

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 MAN key to resume grinding.

5.0 OPERATION

5.08 *Removing, Emptying, and Cleaning the Grinding Vial*

At the conclusion of the grinding cycle, unlatch the lid and lift it. Do not force the lid, but bring it up at its own speed. Open the coil gate by pulling the coil gate handle to the right.

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A reminder: wear cryogenic gloves when handling chilled vials.

Remove the vial from the coil, using gloves or the 6754 Extractor. At this point you may want to shut the lid to conserve liquid nitrogen, or begin another grinding cycle. If the next vial is not pre-cooled, place it in the coil, shut the lid, confirm that a pre-cooling period is programmed, and press RUN to begin the grinding cycle. If you are moving a pre-cooled vial into the coil, shut the lid, program T3 to zero (eliminating the pre-cooling period) and press RUN; grinding will begin immediately.

To open the vial, slip the open end of the 6754 Extractor 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. Always be careful when you do this that the vial does not fall and spill its contents.

Note: ensure that the extractor is clean and dry, especially the threaded end, before using.

If the extractor sticks when removing a cold end plug, this is probably due to water on the extractor screw, which freezes when it contacts the 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 6814 Auto-Extractor (page 22), an optional accessory for the 6750 and 6850 Freezer/Mills.

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 blunt 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 by way of condensation. Samples which were tacky, squishy, 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.

If some condensation on a cold sample is unacceptable, you can 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.

5.0 OPERATION

5.08 *Removing, Emptying, and Cleaning the Grinding Vial*

(Cont'd)

The 6751 Vial may be cleaned quickly and easily by running hot water over it. If it is cold a coating of ice can form on the steel parts, but will melt quickly as the water runs. If sample adheres to the steel parts of the vial, they can be further cleaned with water and soap or detergent, or even with organic solvents. The steel components can also be autoclaved, but if they are to be stored they should be carefully dried first. They are made of magnetic stainless steel, which is corrosion-resistant but will rust if left in contact with water for long periods.

The plastic center cylinder (see page 20) is made of polycarbonate. While this polymer is very tough at low temperatures, it is sensitive to alcohol and organic solvents, and should be cleaned only with soap and water. A mild bleach solution will control organic contamination. Polycarbonate can also be autoclaved, but may be weakened over time by this procedure. While these cylinders are remarkably durable, *always inspect them for cracks before use.*

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Rarely a flanged end plug will stick in the vial when cold. If this happens do not force the end plug out, as you may damage the vial and/or the extractor. The vial can be allowed to warm up gradually, or a hair-dryer or other heat source can be used to warm up the stuck end-plug rapidly. The blunt end-plug can be difficult to remove, even from warm vials. Running hot water over that end of the vial will make it easy.

5.09 ***Checking the level of liquid nitrogen***

Every time you remove or insert a vial, note the level of liquid nitrogen in the tub. You should top it off after every sample. If your programmed grinding cycle includes more than fifteen minutes of actual grinding time, you may wish to visually check the nitrogen level at about that point and add more if needed. We do not recommend cumulative T1 time of more than twenty minutes, as after twenty minutes of grinding time the LN level is close to the point where the LN sensor will shut down the mill and abort the grinding program. If in the operation of your Freezer/Mill you find the LN sensor triggered by a shorter program, keep your programs below that amount of grinding time.

5.10 Optional 6814 **AUTO/EXTRACTOR**

The 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 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. Out of the box, the AUTO/EXTRACTOR is set up for 6801 vials, but it includes an easily installed insert for handling 6751 vials.

5.10 Optional 6814 **AUTO/EXTRACTOR (Cont'd)**

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 6750 FREEZER/MILL, please call SPEX CertiPrep at 1-800-522-7739 or 732-549-7144, Extension 465.

6.0 MAINTENANCE

The 6750 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. When the unit 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 unsightly gunk over the life of the unit.

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If you have spilled any sample powder or liquid inside the unit during a sample run, wait until you are no longer going to continue using the unit, 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. Please Note: After every period of use, the mill is going to be wet with condensation since the Freezer/Mill will pick up moisture as long as it is cold. To maintain the exterior of the unit, first disconnect the Freezer/Mill. Then an occasional spraying and wiping down with a mild window cleaner or similar product will be sufficient. In the rare case that you may need to service the 6750 Freezer/Mill, please call SPEX SamplePrep Customer Service for assistance.
Maintaining the Grinding Vial Sets and the Extractor is basically the normal post-use cleanup covered on pages 17 & 18 - Removing, Emptying, and Cleaning the Grinding Vial.

7.0 ***Accessories: Vials, Vial Opener, Racks, and Extractors***

(If not included in your order, they are available for purchase at additional cost)

6751 Grinding Vial Set

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

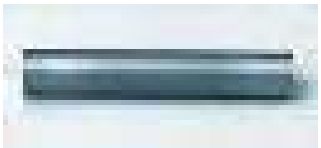
6751C4 Polycarbonate Center Cylinder for 6751 vial set is sold in units of 4. It is also available in units of 20 (part number 6751C20). NOTE: Polycarbonate is durable and transparent but it should not be cleaned with organic solvents, including alcohol.



6751 Grinding Vial Set



End Plug



Impactor



Center Cylinders

A complete 6751 Vial consists of two steel end-plugs (6751E), a steel rod-shaped impactor (6751P), and a polycarbonate tube. The vial is typically assembled by inserting the blunt end-plug into the tube, placing the impactor and sample in the tube, and inserting the flanged end-plug. The flanged end-plug is shaped so that it can be gripped by the 6754 Extractor and Vial Opener, and this end-plug should always face out when a vial is placed in the coil.

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The standard 6751 Vial can be converted to an all-steel vial by substituting **a 6752 Steel Center Section** for the 6751C polycarbonate tube.



6752 Steel Center Section

7.0 ***Accessories: Vials, Vial Opener, Racks, and Extractors***

(If not included in your order, they are available for purchase at additional cost)

The **6753 Microvial Set** consists of the standard 6751E end-plugs, a 25048 polycarbonate tube with holes drilled in it for circulation of liquid nitrogen, and three 6753V Microvials, each with its own steel end-plugs (25049), impactor (37582), and tube (25050). The Microvial Set is used for samples on the order of several hundred milligrams



6753 Microvial Set

The **6754 Extractor** is a dual-purpose tool for opening chilled vials, and transporting them in and out of the Freezer/Mill. One is supplied with every Freezer/Mill



6754 Extractor

NOTE: The open end of the **6754 Extractor** fits over the flanged end-plug of a vial, and the screw threads into the hole in the end-plug (see page 17).

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



6900 Cryogenic Gloves
(Available in Medium, Large, X-Large)

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The **6755 Vial Rack** is a Glass-reinforced acetal rack. This rack holds up to sixteen 6751 vial sets for storage and handling. One is supplied with each 6750 Freezer/Mill and with the 6800B and 6800C Accessory Packages for the 6850 Freezer/Mill.



7.0 ***Accessories: Vials, Vial Opener, Racks, and Extractors***

(If not included in your order, they are available for purchase at additional cost)

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 or 732-549-7144 Ext. 465**

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8.0 *PARTS LIST & ITEM LOCATOR*

(see Diagrams A, B, & C)

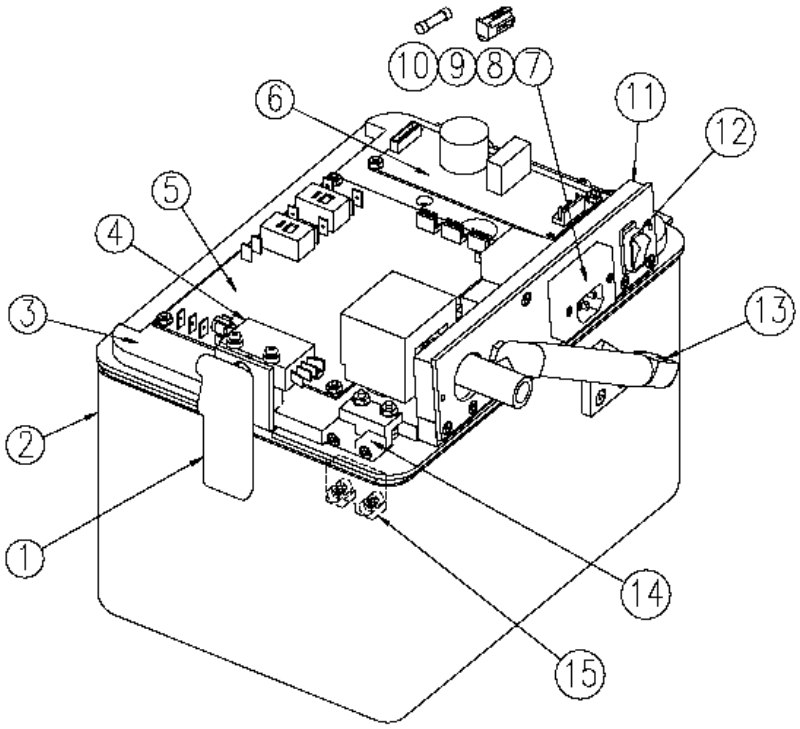
Item	Part No.	Description	Location (Diagram, Page #)
1	50587	Latch	A, 24
2	38550	Tub, Outer	A, 24
3	39079	Foam, Top Plate	A, 24
4	92679	Switch, Pushbutton	A, 24
5	38569	Driver Board	A, 24
6	38579	Power Supply	A, 24
7	96004	AC Inlet	A, 24
8	96005	Fuse Holder	A, 24
9	93850	Fuse, 8 Amp for 115V (2 required)	A, 24
10	93840	Fuse, 5 Amp for 230V (2 required)	A, 24
11	39080	Foam, Back-plate	A, 24
12	92703	Power Switch	A, 24
13	38567	Gas Spring for Lid	A, 24
14	92710	Switch, Magnetic	A, 24
15	92711	Actuator, Magnetic	A, 24
16	50588	Hinge	B, 25
17	38531	Top Cover	B, 25
18	38562	Processor Board	B, 25
19	38549	Control Panel Overlay	B, 25
20	39043	Foam, Sealing Plate	B, 25
21	24456	Tub, Inner	B, 25
22	31522	Collar	B, 25
23	38560	Tub Insulation	B, 25
24	93725	Rubber Foot (4 Required)	B, 25
25	24445	Anvil	C, 26
26	50541	Retaining Ring	C, 26
27	32350	Rod	C, 26
28	38853	Liquid Level Sensor	C, 26
29	38539	Pivot Pin Yoke	C, 26
30	38548R	Coil (Solenoid) Assembly, 115V/230V	C, 26

NOTE: Please refer to pages 20 and 21 for vial accessory parts.

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9.01 DIAGRAM "A"

(View from rear of unit w/cover removed for clarity)

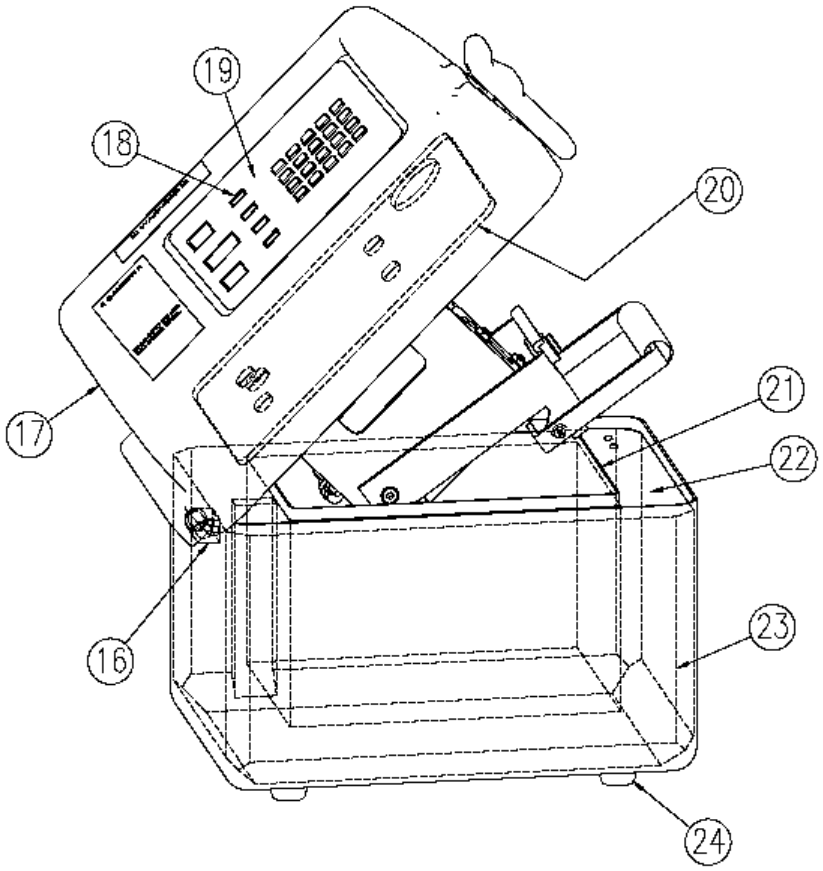


Item	Part No.	Description
1	50587	Latch
2	38550	Tub, Outer
3	39079	Foam, Top Plate
4	92679	Switch, Pushbutton
5	38569	Driver Board
6	38579	Power Supply
7	96004	AC Inlet
8	96005	Fuse Holder
9	93850	Fuse, 8 Amp for 115V (2 required)
10	93840	Fuse, 5 Amp for 230V (2 required)
11	39080	Foam, Back-plate
12	92703	Power Switch
13	38567	Gas Spring for Lid
14	92710	Switch, Magnetic
15	92711	Actuator, Magnetic

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9.02 DIAGRAM "B"

(view from rear without top cover of lid)

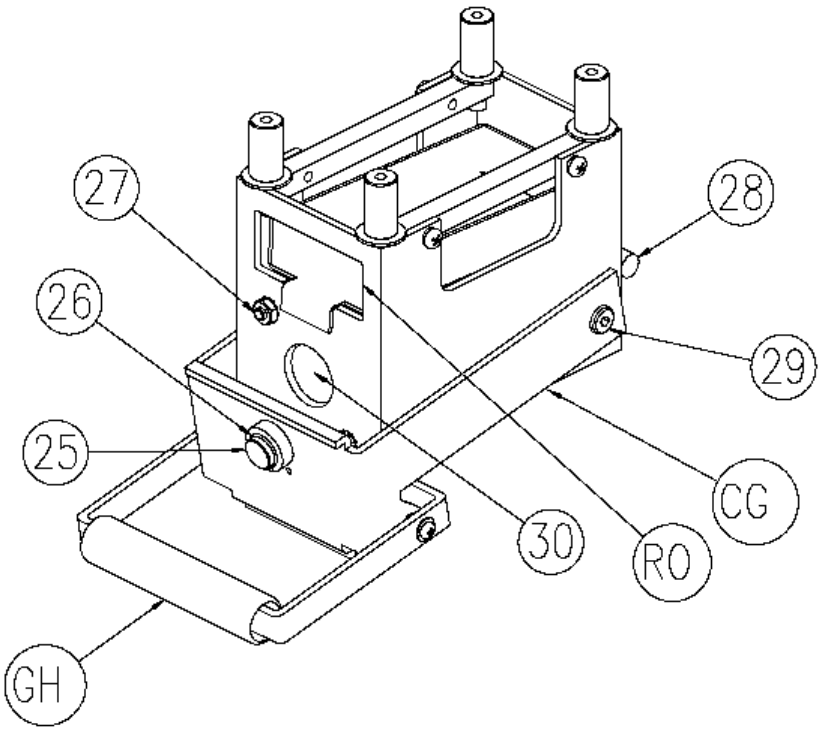


Item	Part No.	Description
16	50588	Hinge
17	38531	Top Cover
18	38562	Processor Board
19	38549	Control Panel Overlay
20	39043	Foam, Sealing Plate
21	24456	Tub, Inner
22	31522	Collar
23	38560	Tub Insulation
24	93725	Rubber Foot (4 Required)

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9.03 DIAGRAM "C"

(Magnetic Coil Assembly)



Item	Part No.	Description
25	24445	Anvil
26	50541	Retaining Ring
27	32350	Rod
28	38853	Liquid Level Sensor
29	38539	Pivot Pin Yoke
30	38548R	Coil (Solenoid) Assembly, 115V/230V

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10.0 TROUBLE-SHOOTING GUIDE

	PROBLEM	CAUSE	SOLUTION
1	The unit does not turn on	No power	Make sure power cord is plugged into outlet and inlet
2	The unit does not turn on	No power	Check outlet for power and correct as required
3	The unit does not turn on	Power switch not turned on	Switch unit on
4	The unit does not turn on	Blown fuse	Replace fuses
5	The unit does not turn on	Blows fuses repeatedly	Electrical short (**RTF**)*
6	LED displays “Lid” and “Fault”	Safety interlock switch not engaged	Close the lid and latch
7	LED displays “Lid” and “Fault”	Lid is not closed completely	Push the lid closed and latch into place
8	LED displays “LN” (When RUN is pushed)	Liquid nitrogen sensor senses inadequate liquid nitrogen level	Pour more liquid nitrogen into the tub
9	Coil gets very warm	Low liquid nitrogen levels	Pour more liquid nitrogen into the tub
10	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
11	Impactor doesn’t shuttle back and forth	Impactor is magnetized	Remove impactor, turn end-for-end, or demagnetize impactor and re-insert
12	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
13	Flanged End Plug doesn’t come off cold vial.	End plug/tube joint too tight when very cold.	Allow vial to warm up

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

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10.0 TROUBLE-SHOOTING GUIDE (cont'd)

	PROBLEM	CAUSE	SOLUTION
14	Blunt end-plug doesn't come off room-temperature vial	Hard to grip	Warm vial under hot water. Use wide-jaw pliers if necessary.
15	Manual extractor jams	Water on extractor screws or bell	Dry extractor before each use
16	Manual extractor jams	Ice in threaded hole in end plug	Dry end plug thoroughly before using
17	Plastic center cylinder cracks	Cylinder cleaned with alcohol or other organic solvent	Use bleach, detergent, and water to clean cylinders
18	Plastic center cylinder cracks	Vial assembled when chilled	Never force end-plugs into cold cylinder
19	Plastic center cylinder cracks	Sample piece acts as wedge	Reduce size of sample pieces. Make sure there are no sample particles on cylinder ends.

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

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11.0 WARRANTY

SPEX SamplePrep LLC 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 SamplePrep LLC carry the manufacturer's warranty only.

11.01 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.

11.02 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.

To Contact Us

SPEX SamplePrep LLC
203 Norcross Ave.
Metuchen, New Jersey 08840

Tel.: 732-549-7144 or 800-522-7739
Fax: 732-906-2492
Website: www.spexcsp.com
E-mail: sampleprep@spexcsp.com

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