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5100 MIXER/MILL[®]

OPERATING MANUAL

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1.0 INTRODUCTION

The SPEX CertiPrep 5100-series Mixer/Mills are efficient, compact laboratory mills capable of pulverizing samples in the 1-gram range, or mixing product samples with volumes to 3ml. The clamp can hold three ½ inch diameter vials, or two ¾ inch diameter vials, with length up to 2 inches.

Functionally described as shaker mills or high-energy ball mills, these Mixer/Mills shake containers back and forth thousands of times a minute. Multi-purpose mills, they are capable of rapidly reducing hard, brittle samples to analytical fineness, blending powders, and making emulsions.

The vial, which contains a sample and one ball, is shaken in a complex motion that combines back-and-forth swings with short lateral movements, each end of the vial describing a figure-8. The length of that swing is about one inch. With each swing the ball impacts against one end of the vial, simultaneously milling the sample to a powder and blending it. Because of the amplitude and velocity of the clamp's swing, each ball develops fairly high G-forces, enough to pulverize the toughest rocks, slag and ceramics.

The single-clamp 5100 Mixer/Mill, has served spectroscopists and analytical chemists for over forty years. The clamp mechanism has changed little since the earliest versions, simply because it has proven extremely durable. Many of the earliest 5100 Mixer/Mills are still in service. An electronic 100-minute timer has replaced the original wind-up timer and the mill's safety features have been improved.

5100-series Mixer/Mills have been used for pulverizing rocks, minerals, sand, cement, slag, ceramics, catalyst supports, and hundreds of other brittle, often hard samples. Early on, the 5100 was used to grind samples, then blend them with graphite for arc/spark spectroscopy or to mix samples with KBr to be pressed into 13mm wafers for IR analysis. Today the 5100 is still being used for dozens of small-scale mixing and milling tasks in the lab.

2.0 SPECIFICATIONS

Type of mill: grinding mixer/mill

Grinding Containers: grinding vial with ball

Weight (empty, without vial): 15.2 lbs. (7 Kg)

Dimensions: 12 in (33 cm) x 8 in (20 cm) x 8 in (20 cm)

Grinding Vials Available:

3112	Transparent Methacrylate Balls (3/8")
3119	Transparent Methacrylate Balls (1/8")
3111	Polystyrene Vial 1/2 x 1" lg.
3116	Polystyrene Vial 1/2 x 2" lg.
3116PC	Polycarbonate Vial 1/2 x 2" lg.
6133	Polystyrene Vial 3/4 x 2" lg.
3114	Stainless Steel Vial Set
3117	Hardened Tool Steel Vial Set
3127	Hardened Tool Steel Vial Set
3118	Agate Vial Set
5004	Tungsten Carbide-Lined Vial Set

Note: in practice, the sample capacity of any particular vial depends on the properties of the sample, particle-size requirements, and other considerations.

Electrical specifications: available in 115 60Hz or 230V 50Hz.

Meets European CE Directives

Cord: 115V 60HZ version, 3-prong grounded cord supplied.
230V 50HZ version, with CEE 7/7 two-prong plug supplied.

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

Fuses: 115V 60HZ version, 4-amp slow-blow fuse in the power inlet module

230V 50HZ version, 2-amp slow-blow fuse in the power inlet module

Motor: 1/15 Hp, 3000 RPM (115V), 2500 RPM (230V/50Hz)

Timer: 1 minute to 30 minutes (115V)

1 minute to 72 minutes (230V)

3.0 UNPACKING

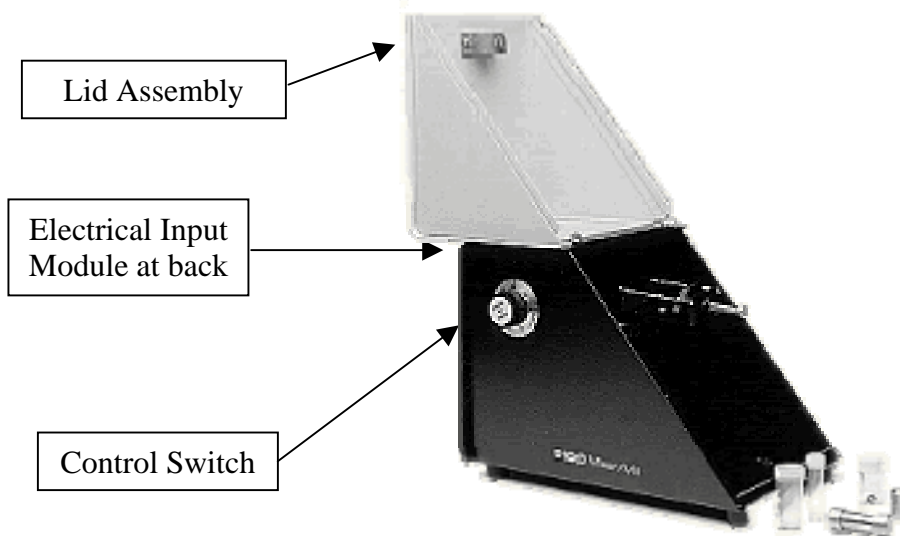
Any visible damage to the shipping container should immediately be reported to the carrier. If there is no visible damage, remove all packing documents from the exterior of the box, and after completion of your inspection, file in your records.

Open the top of the shipping box. Remove the foam packing material. Carefully grasp the mill from both sides, exercising caution while using proper lifting techniques to remove the unit from the carton (although small, the unit weighs 15 lbs). Place the mill on the bench top where it will likely stay, and open the lid. Look for any hidden damage that may have occurred during shipping. Save the box for possible future use.

Follow a logical sequence of steps as you inspect the unit (see Figure 2). For example:

1. Inspect the outside of the cabinet for any visible damage.
2. Are any of the switches damaged?
3. Is the latch mechanism damaged?
4. Inspect the interior of the mixer.
5. Is the motor damaged?
6. Do all the springs and mounting points seem okay?
7. Inspect the accessory pack. Compare with the packing list.

If everything seems to be okay, proceed to the next section “ SETTING UP.”



5100 Mixer/Mill

(Figure 1)

4.0 SETTING UP

Remove the plastic bag containing the instructions and spare fuses. Open the lid and remove the packing from around the clamp. Loosen the locking nut on the clamp, and unscrew the clamp a few turns.

BEFORE OPERATING, REMOVE THE SHIPPING BOLT FROM THE BOTTOM OF THE CASE OR YOU MAY DAMAGE THE MIXER/MILL. THIS SCREW SHOULD BE REPLACED IF THE INSTRUMENT IS EVER TO BE RESHIPED.

The #5100 has a high-speed motor, which requires the voltage to be close to that stated. Low voltage may cause the motor to rotate at a low speed and ultimately damage it. The instrument is designed to operate at normal room temperatures. Too low a temperature will again result in low speed. If, on the other hand, the motor overheats, a built-in thermal overload switch will stop it automatically at 120 C, and will permit resumption of operation after the motor is cool.

4.01 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. The mill uses 4-amp "slow-blow" fuses. The 230 V 50 HZ model uses 2-amp "slow-blow" fuses and comes with a European-style polarized plug; outside of Europe the cord should be adapted to local requirements.

5.0 OPERATION

After inserting the sample that you are going to grind or mix, into the vial set, place the vial into the clamp. Tighten the clamp with the knob, and lock the clamp with the wing nut. If you are working with one vial, place it in the center of the clamp. If you have two vials, space them evenly, one towards each side of the clamp. The 5100 Mixer/Mill's clamp will also hold three ½-inch-diameter vials, one in the center and one on each side of the clamp. Close the lid and fasten down the lid with the manual latch.

Adjust the timer setting as described below under **Timer**.

5.01 TIMER

The timer of the 5100 Mixer/Mill operates for a maximum of 30 minutes (115V/60Hz model) or 72 minutes in the case of the 230V/50Hz model. The timer is set by lining up the desired dial setting with the mark on the case. To be sure of the mark, rotate the timer dial counterclockwise until it stops; this is the zero position and the timer mark on the case is adjacent to the zero on the timer dial. To set the timer, rotate the dial clockwise from the zero position to the desired setting.

5.01 TIMER
(Continued)

To start the mill, push in the white button in the center of the dial (the button marked “push to start”). This initiates the timer sequence. If this button is pushed during a run, it will not stop the mill but it does start the timing period over.

Once set, the timer will reproduce running times within a second or two. (This timer dial does not move counterclockwise to indicate elapsed time, or have to be reset after every run.) Some technicians tape the dial in place so the setting is not changed accidentally.

In the case of very short run times (under one minute) we recommend verifying the timer setting with a stopwatch.

To remove the vial, loosen the locking tab, then open the clamp.

5.02 THE CLAMP

The 5100 Mixer/Mill has a tray-type clamp which adjusts to hold vials from 1 inch to 2-1/8 inches in length, and up to 3/4 inches in diameter. It can hold, side by side, either three 1/2 inch diameter vials or two 3/4 inch diameter vials.

The clamp is loosened and tightened by a thumbscrew attached to the moveable jaw of the clamp. When using one vial, always center it in the clamp, because the moveable jaw can pivot and loosen if the vial is off center. Once tightened, the clamp screw is secured by a wing nut, which should be screwed tight against the end of the clamp.

The 5100 Mixer/Mill is robust but we do not recommend running it routinely with loads of more than 45 grams, sample(s) and vial(s) combined weight.

5.03 MIXING AND GRINDING

Mixing is usually done in a plastic vial with plastic balls. Depending on the nature and amount of material to be mixed, one or several balls may be used. With a small amount of material and more than one or two balls, the impact may break the bottom of the plastic vial, so keep the number of balls to a minimum. Plastic vials and balls can also be used to grind soft metals (e.g. antimony), carbon, and other easily friable materials.

For samples that cake during mixing, a slurry with water or alcohol may be helpful, if compatible with the vial. Water may be dried out afterwards by heating plastic vials in a very low temperature oven. If caking is due to static charges, a small amount of graphite can be added to the vial.

Grinding is done in the metallic and ceramic containers: steel, tungsten carbide, stainless steel, and agate. Dry grinding is the simplest approach and is most often used. The criteria for container selection are usually those of grinding efficiency vs. contamination; steel and tungsten carbide grind more rapidly than agate, but contaminate more.

5.0 OPERATION

5.04 MIXING

In general, samples may be mixed in about 15 seconds using a 3/8" diameter (#3112) ball in either the 1" or 2" vial. For very small amounts of sample, one or two 1/8" diameter methacrylate balls (#3119) will be found more suitable.

For mixing certain fluffy or light materials, the addition of a drop of alcohol is recommended.

Many brittle substances such as platinum sponge, bismuth and antimony metals may be ground in the plastic vials.

For those materials which have a strong tendency to pack, such as asbestos fibers, reduce the sample size to a minimum and use two balls.

5.05 GRINDING

Stainless steel, hardened steel, and agate vials are available for grinding hard material. Of these agate is the hardest, is finished to the best polish, and will best resist the corrosion of most chemicals. In general, the vial should be filled to a maximum of about 1/3 its capacity. The smaller the sample size, the shorter the grinding time will be.

For the KBr technique of infrared analysis, the agate or stainless steel vial is recommended. Although plastic vials will grind and mix the KBr and sample adequately, contamination from the plastic will result in several extraneous bands (see the SPEX SPEAKER, Vol. 11, No 4, 1957: Applied Spectroscopy, 14, 135,1960). Whether these bands are objectionable depends on the problem and will have to be determined by prior experiment.

While expensive, the agate vial is best for the KBr technique. Because of the high polish of the walls, there is little chance of entrapment of the sample.

In the KBr technique, the minimum grinding time should be employed in order to avoid hydroxyl contamination. These bands sometimes appear after long periods of grinding and are due to moisture.

A typical mixing load is about 40% of the vial's volume. If a sample is not being ground fine enough, one can a) decrease the amount of sample, b) increase grinding time, c) add a grinding aid or a liquid. Wet grinding keeps the sample from caking and will give a smaller final particle size, but there are handicaps: 1) an extra drying step is required; 2) the fluid used for wet grinding must be chosen carefully not to alter the sample or attack the container; 3) not all the vials are leak proof. The 5004 tungsten carbide vial and all plastic vials are more or less watertight. The 3114 and 3117 steel vials can hold water with the help of the 51705 O-ring.

5.0 OPERATION (Continued)

Avoid grinding loads that are too small in brittle containers such as agate. If there is not enough material to cushion the impact of the balls, they will generate excess contamination and can in extreme cases chip the container.

Caution: always use grinding balls that match the material of the container, e.g. steel balls for a steel container, agate balls for an agate container, etc. This will limit contamination. An inappropriate choice of balls, such as tungsten carbide balls in an agate container, can damage the container.

CAUTIONARY NOTE for #3118 Agate

The construction of the #3118 is such that excessive tightening of the tapered plug will break the walls of the container. For this reason, it is necessary to place the unit in a plastic vial (our #6133 is supplied) and then hold the latter in the clamp of the Mixer/Mill. Foam rubber bumpers are used at both ends of the vial to secure it in place. Because the tapered plug may jiggle against the foam rubber and permit seepage during grinding, it is advisable to run a piece of tape over the plug and along the side of the vial.

5.06 MULLS

Mineral oil mulls may be made using the plastic or steel vials. The possibility of plastic contamination in infrared analysis should still be considered.

6.0 Accessories: Grinding/Mixing Vials

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

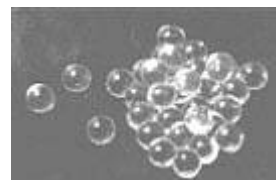
3112 Transparent Methacrylate Balls

3/8 in. diameter; fit all SPEX CertiPrep plastic vials. Sold in units of 100.



3119 Transparent Methacrylate Balls

1/8 in. diameter; fit all SPEX CertiPrep plastic vials. Sold in units of 100.



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

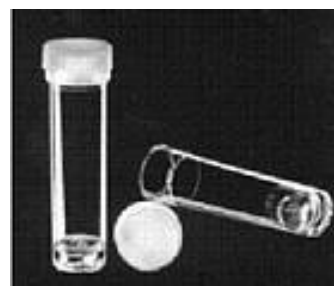
3111 Polystyrene Vial

1/2 in. diameter x 1 in long. Includes slip-on polyethylene cap; capacity 2.5ml; mixing load approximately 1ml; accepts SPEX CertiPrep 3112 or 3119 plastic balls. Sold in units of 100.



3116 Polystyrene Vial

1/2 in. diameter x 2 in. long. Includes slip-on polyethylene cap. Capacity 5 ml; mixing load approx. 2 ml; accepts SPEX CertiPrep 3112 or 3119 plastic balls. Sold in units of 100.



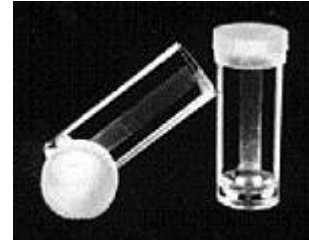
To Order any of these accessories, call Customer Service at 800-522-7739 or 732-549-7144

6.0 Accessories: Grinding/Mixing Vials

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

6133 Polystyrene Vial

3/4 in. diameter x 2 in. long. Includes slip-on polyethylene cap. Capacity 12ml; mixing load approximately 5ml; accepts SPEX CertiPrep 3112 or 3119 plastic balls. Sold in units of 100.



3114 Stainless Steel Vial Set

1/2 in. diameter x 1 in. long. Made of 303 stainless steel; includes slip-on cap and 1/4 in. steel ball; grinding load 0.2-0.6 ml, mixing load 1ml.



3117 Hardened Tool Steel Vial Set

1/2 in. diameter x 1 in. long. Made of Worpliss tool steel. Includes slip-on cap and 1/4 in. steel ball. Grinding load 0.2 – 0.6 ml, mixing load 1 ml. Four balls per set.



3127 Hardened Tool Steel Vial Set

3/4 in. diameter x 1 7/8 in. long. Made of Worpliss tool steel; includes center cylinder with two slip-on caps and 1/4 in. steel ball. Grinding load 0.5–1.0 ml, mixing load 2 ml.



6.0 Accessories: Grinding/Mixing Vials

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

3118 Agate Vial Set

Handmade from flawless Brazilian agate; must be run inside cushioned SPEX CertiPrep 6133 vial (included); includes stopper and 1/4 in. agate ball. Grinding load 0.2-0.6ml, mixing load 1 ml.



5004 Tungsten Carbide-Lined Vial Set

3/4 in. diameter x 2 1/8 in. long. Includes two slip-on Delrin® caps with tungsten carbide inserts, six disposable methacrylate center cylinders, and two 5/16 in. tungsten carbide balls. Grinding load 0.5-1.5ml; mixing load 3ml.



5004A Tungsten Carbide-Ball Set
5/16 in. diameter, for SPEX CertiPrep 5004 vial. Four balls per set.

5004C Methacrylate Center Cylinder
For SPEX CertiPrep 5004 vial, sold in units of 20.

To Order any additional accessories, call Customer Service at 800-522-7739 Ext. 465

7.0 MAINTENANCE

The 5100 MIXER/MILL is designed to be virtually maintenance-free. Both the motor and main ball bearing are sealed and lubricated for life.

The size of the jaw opening makes it difficult to overload, and consequently damage, the instrument. However, do not shake anything above 45 grams in weight, INCLUDING CONTAINER.

In order to assure proper performance, perhaps the most important factor is overall cleanliness. Any spilled powders or liquids should be wiped up immediately with a damp cotton cloth. This should also be done occasionally after extended periods of use. This should minimize the buildup of any powders, mold/mildew, or unsightly gunk over the life of the unit.

To maintain the exterior of the unit, first disconnect the 5100 MIXER/MILL, then an occasional spraying and wiping down with a mild window cleaner or similar product will be sufficient.

NOTE: the mill should be disconnected from power source FIRST, before any cleanup or maintenance work is to be performed on the mill.

The threads of the clamp screw should be kept clean to avoid jamming. The foam pads in the clamp should be replaced when damaged, worn, or missing.

The 5100 Mixer/Mill is intended for intermittent use with running times that are typically 5 to 20 minutes long. Under those conditions it should run for many years without maintenance other than that described above.

SPEX warrants the 5100 Mixer/Mill against defects of materials and workmanship for a period of one year from date of shipment. There is no warranty against breakage for tungsten carbide and agate vials.

If you have any questions about the operation and maintenance of your 5100 Mixer/Mill, please contact our specialists by phone at 1-800 LAB-SPEX (522-7739) or 732-549-7144, or by e-mail at sampleprep@spexcsp.com.

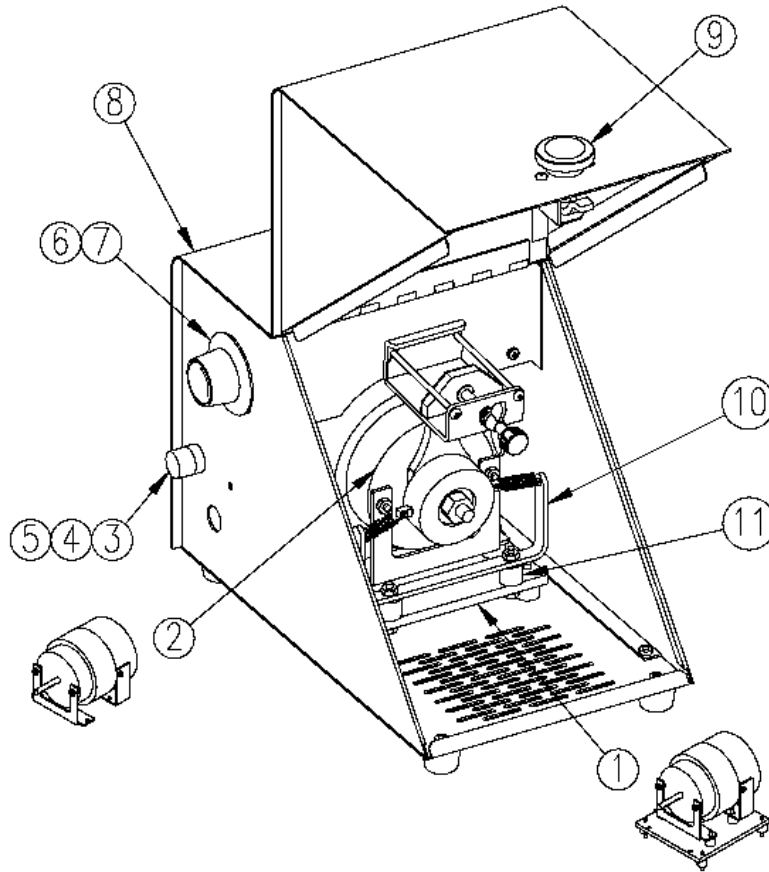
8.0 PARTS LIST & ITEM LOCATOR

(See Diagram A, B, & C)

<u>Item</u>	<u>Part No.</u>	<u>Description</u>	<u>Location</u> (Diagram, Page #)
1	5100-2	MOTOR, BASE PLATE, AND BRACKETS	A, 16
2	30215	MOTOR ASSEMBLY	A, 16
3	93890	FUSE HOLDER	A, 16
4	93830	FUSE, 4 AMP (115V)	A, 16
5	93810	FUSE, 2 AMP (230V)	A, 16
6	53170	PUSH BUTTON TIMER, 30 MIN (115V)	A, 16
7	53171	PUSH BUTTON TIMER, 72 MIN (230V)	A, 16
8	24732	CABINET	A, 16
9	50523	LATCH	A, 16
10	24290	SPRING SUPPORT BRACKET	A, 16
11	50152	VIBRATION MOUNT	A, 16
12	22747	PAD FOR MOVING CLAMP	B, 17
13	5100-8	CLAMP (INCLUDES 5100-9)	B, 17
14	5100-8A	CLAMP ASSY (INCL. 22754, 50015, 5100-9)	B, 17
15	22746	PAD FOR FIXED CLAMP	B, 17
16	22749	MOVABLE CLAMP FACE	B, 17
17	5100-9	THUMB SCREW ASSEMBLY	B, 17
18	61750	WING NUT	B, 17
19	22750	THUMB SCREW	B, 17
20	52109	SPRING (2 REQUIRED)	B, 17
21	22754	ECCENTRIC	B, 17
22	50015	BEARING	B, 17
23	21609	SPADE BOLT (2 REQUIRED)	B, 17

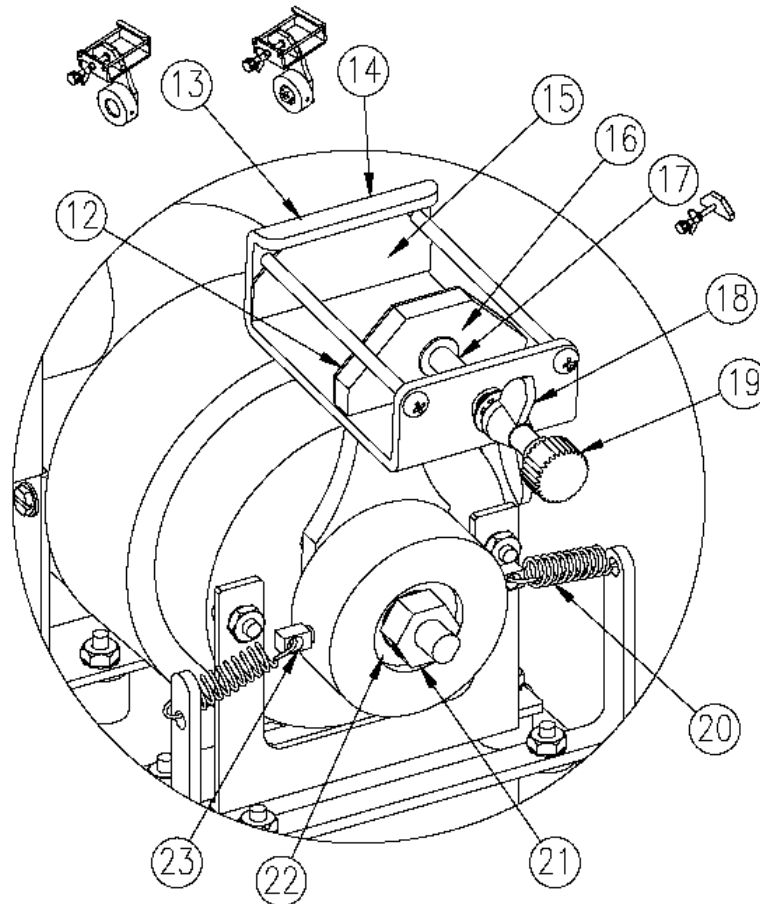
9.0 DIAGRAM "A"

(Shown without internal cover plate)



<u>Item</u>	<u>Part No.</u>	<u>Description</u>
1	5100-2	MOTOR, BASE PLATE, AND BRACKETS
2	30215	MOTOR ASSEMBLY
3	93890	FUSE HOLDER
4	93830	FUSE, 4 AMP (115V)
5	93810	FUSE, 2 AMP (230V)
6	53170	PUSH BUTTON TIMER, 30 MIN (115V)
7	53171	PUSH BUTTON TIMER, 70 MIN (230V)
8	24732	CABINET
9	50523	LATCH
10	24290	SPRING SUPPORT BRACKET
11	50152	VIBRATION MOUNT

9.1 DIAGRAM "B"



<u>Item</u>	<u>Part No.</u>	<u>Description</u>
12	22747	PAD FOR MOVING CLAMP
13	5100-8	CLAMP (INCLUDES 5100-9)
14	5100-8A	CLAMP ASSY (INCL. 22754, 50015, 5100-9)
15	22746	PAD FOR FIXED CLAMP
16	22749	MOVABLE CLAMP FACE
17	5100-9	THUMB SCREW ASSEMBLY
18	61750	WING NUT
19	22750	THUMB SCREW
20	52109	SPRING (2 REQUIRED)
21	22754	ECCENTRIC
22	50015	BEARING
23	21609	SPADE BOLT (2 REQUIRED)

11.0 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.

11.1 Product Specifications

Every effort has been made to provide complete and accurate product operation and 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.

11.2 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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Metuchen, New Jersey 08840

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E-mail: sampleprep@spexcsp.com
Customer Service: 800-522-7739 Ext. 465