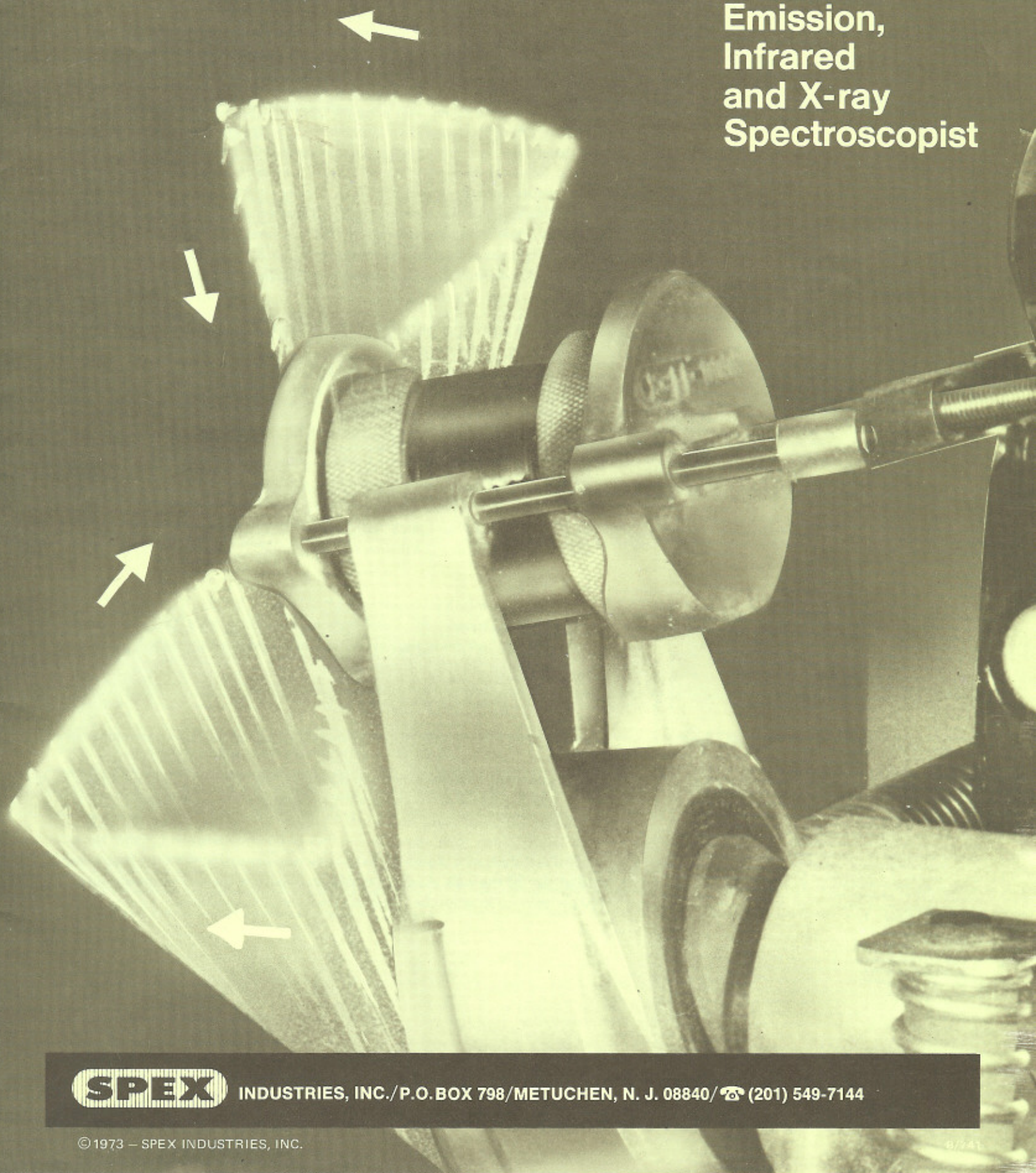


Sample Preparation

for the
Emission,
Infrared
and X-ray
Spectroscopist



SPEX

INDUSTRIES, INC./P.O. BOX 798/METUCHEN, N. J. 08840/☎ (201) 549-7144

Introduction

To prepare samples for the multitude of analyses conducted in today's laboratories, techniques have proliferated almost as fast as analytical methods themselves. Spex Industries has earned the reputation for providing quality products to perform the blending, diluting, fusing, grinding, and briquetting operations prior to analysis by emission, infrared, and x-ray spectroscopy as well as wet chemistry. Conceived by and for spectrochemists our devices do what is expected of them reliably and efficiently. Prior to distribution to analysts throughout the world, lunar rocks were ground in our grinders; 15,000 specially cleaned 3111, 3116 and 6133 plastic vials were the storage containers.

The first step in any analytical procedure is to obtain a representative sample whether from blast furnace slag, a moon rock, an ore body, a batch of plastic pellets, Portland cement, a bone, a pharmaceutical—or any of thousands of other natural or man-made products. Ideally, it must be small enough for convenient analysis yet compositionally representative of the total material. Generally, this sampling operation involves progressive reduction of that material from good-size chunks down to 100 grams or less of a fine powder. At the same time the particle size is reduced, the sample is continuously blended to prevent sampling bias.

The type of sampling preparation device needed depends on:

- the friability of the material;
- the amount of material to be ground and analyzed;
- the tolerance of the analysis to certain contaminants which may be introduced in the grinding operation;
- the number of samples to be ground and the production rate demanded.

Thousands of customers throughout the world have concluded that the most universally applicable devices for the purpose are high-speed impact and swing mills. Inside a vial made of materials as diverse as tungsten carbide and polystyrene a ball or puck of the same material is reciprocated or "hula-hooped" against the substance to be ground.

Cover Photo

Complex and violent, the agitation may be represented by components in three mutually perpendicular directions. For the 8000 MIXER/MILL the main component is a swing through about 2½" at the end of a 3½" arm, an arc of approximately 40°. Within a single cycle, there is an additional vertical component of 3/16" and horizontal of 9/16". This movement is repeated some 1200 times per minute. In the 5100 MIXER/MILL, the three-dimensional motion is scaled down to an action consistent with the shape and smaller size of the vials. At every stroke an impact at the end of the vial—over 100 per second—crushes the material rapidly and reproducibly.

Contamination

Practically speaking, the best way to avoid contamination while grinding is to choose a grinding medium the composition of which will not adversely affect the final product or analysis. From the standpoint of minimizing container pickup, tungsten carbide and agate are the best materials. One example is Portland cement ground in the Shatterbox for a normal one minute prior to x-ray analysis. With the 8501 hardened steel container, 0.15% iron was picked up; with the 8504 tungsten carbide container only 0.02% was picked up.

An extensive paper on contamination (Geoffrey Thompson, Donald C. Bankston, App. Spec., 24, 210, 1970) is supported by a wealth of data from Woods Hole Oceanographic Institute, showing how much pickup of various elements can be expected when SiO_2 or CaCO_3 is ground. Agate (3118) and methacrylate (8006) were found to contribute almost no contamination.

Cleaning Containers

The most efficient means of grinding large numbers of samples without intersample contamination is to "dry-clean" the container between runs. A small portion of the new sample is ground for a minute or so then discarded.

Overcoming Static

In dry weather materials with a high dielectric constant develop repelling charges and do not mix. "Rinsing" with graphite powder lines the container and impactors with a conductive medium.

Wet Grinding

Water or alcohol in small amounts often facilitates grinding and mixing; but remember that too much alcohol will soften polystyrene. Submicron particle sizes are attained routinely with slurries.

Infrared Mulls

The 3111 polystyrene vial is used by many infrared laboratories for preparing Nujol mulls and KBr pellets. In routine work, the polystyrene spectrum appears as a constant background and is simply subtracted out mentally.

Grinding Aids

Almost everyone lays claim to his own magic potion which, when sprinkled in with the substance to be ground, facilitates grinding primarily by inhibiting caking. X-ray spectroscopists—experts with the Shatterbox—seem to prefer sodium stearate. Household "Tide" and "Boraxo," which contains an abrasive, and "Avicel," a granular cellulose, are free of metallic elements.

No. 8500 Shatterbox®

An extremely fast and efficient grinder, the Shatterbox spins a heavy puck and ring around the inside of a closed dish at 900 rpm. Dishes come in a variety of materials (hardened steel, high alumina ceramic, tungsten carbide) to accommodate all manner of samples. Although the Shatterbox is most efficient when grinding samples occupying 25 ml or more, three sets of grinding vials and racks are available for multiple grinding of smaller samples: the 8503 and the 8507, made of hardened steel; and the WC 8508.



8500 SHATTERBOX WITH 8501 DISH

A quick scan of the material listed below will reveal how universal the Shatterbox is as a tool for grinding production samples quickly and reproducibly prior to x-ray and emission spectrochemical analysis.

A prime application, interesting if unexpected is grinding Portland cements and intermediate raw mixes to maintain uniform high quality concrete production.

In the metals industries, the Shatterbox is helping to stabilize the composition of slags, raw materials and master alloys. Other users are manufacturers of welding fluxes, fertilizer, pesticides, inorganic chemicals as well as research laboratories in the geological and mining fields.

One to four samples will be ground at no charge, a full report and recommendations returned within two weeks. (See P. 15)

Grinding Tests Using 8501 Hardened Steel Container

Material	Form as received	Time, min	Amount grams	% Passing 325 mesh
Asbestos	Fibrous	12	20	100
Cement, Portland raw mix	+60 mesh	2½	40*	100
Ferro-chromium	+100 mesh	5	25	100
Ferro-manganese	+200 mesh	3	25	100
Ferro-molybdenum	-80 mesh	4	25	100
Ferro-niobium	-80 mesh	3	25	100
Ferro-silicon	-80 mesh	4	25	100
Ferro-titanium	-80 mesh	6	25	100
Ferro-vanadium	-80 mesh	7	25	100
Fiberglas	thin sheets	2	10	100
Fluorspar	+100 mesh	3	50	100
Pesticide	-100 mesh	15	50	100
Phosphate, raw mix	+60	2½	40	100
Iron powder	-80	6	5	68
Sand	-10	10	100	100
Slag, blast furnace	chunks	1	10**	100
Slag, open hearth	chunks	1	20	76
Transite	chunks	10	35	100

* sodium alkylarylsulfonate added, 5%

** Household detergent (Tide) added, 10%



8503R HOLDING SEVEN 8503

8507R HOLDING THREE 8507



8507 HARDENED STEEL
8508 TUNGSTEN CARBIDE



8504



8505



8500 Shatterbox® Grinder and blender, 1/3 hp motor, 50-60 Hz, 115V or 230V (specify), 0-15 min timer, ϕ 13" x 20" high, 160 lb, includes 8506 silencer cover (230V supplied without timer or electrical plug).

8503R Rack for holding up to seven 8503

8507R Rack for holding up to three 8507 or 8508

	Material	O.D.	Capacity ml	Recommended load ml	Weight lb
8501	Hardened steel	6½" x 2¾"	360	20-50	18
8502	Hardened steel **	6½" x 2¾"	360	20-50	18
8503	Hardened steel	2-5/8" x 3/8"	30	3-10	2
8504	WC*	7" x 3"	245	25-70	29
8505	Alumina ceramic*	6" x 3"	180	20-60	11
8507	Hardened steel	3¾" x 2¼"	95	10-30	5
8508	WC*	3¾" x 2¼"	95	10-30	8

* Because this material is brittle as well as hard, caution is required to prevent chipping and breaking, against which no guarantee can be offered; with the proper technique (instructions are included) hundreds of laboratories obtain years of grinding service.

** The lid of this dish has gas inlet for grinding under inert atmosphere.

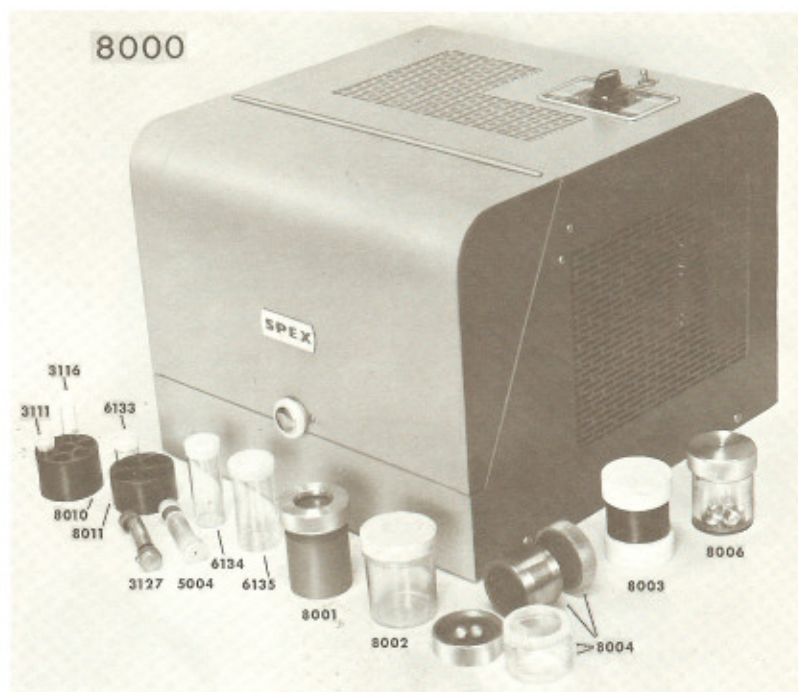
Grinding Containers

No. 8000 Mixer/Mill®

PULVERIZES 10 ml in a single load.
MIXES with plastic vials and balls to avoid metallic contamination.

The entire mechanism is enclosed in an attractive sheet metal case coated with a heavy chemically resistant paint. The hinged door actuates an interlocking safety switch. A timer automatically turns the MIXER/MILL off after any preset time up to one hour. A parallel switch permits operation for longer periods. The instrument is shock mounted so that it may be placed on a table with other apparatus. Noise level, too, is largely quieted by this shock mounting system.

One to four samples will be ground at no charge, a full report and recommendations returned within two weeks. (See P. 15)



8000 Mixer/Mill®, 115V, 60 Hz, for mixing quantities of 10-100 ml, grinding 3-10 ml, 1 hour timer, continuously variable jaws holding vials up to ϕ 2 1/4" x 3 1/4" long, rugged construction and housing, shock mounted, 15" x 16" x 12", 75 lb

220V, 50 Hz model

8010 Adapter for 7 ϕ 1/2" vials

8011 Adapter for 4 ϕ 3/4" vials

Grinding Containers

	Material	O.D.	Capacity, ml	Recommended load, ml	Weight
3111*	Polystyrene	1/2" x 1"	2	1	4 lb/M
3116*	Polystyrene	1/2" x 2"	5	3	7 lb/M
6133	Polystyrene	3/4" x 2"	10	5	14 lb/M
6134	Polystyrene	1" x 3"	30	15	32 lb/M
6135	Polystyrene	1 1/4" x 3"	60	30	36 lb/M
8002	Polystyrene	2-1/8" x 2 1/2"	100	10-50	90 lb
3112	Methacrylate ball	3/8" ϕ			2 lb/M
3114*	Stainless Steel	1/2" x 1"	2	0.2-0.6	1 oz
3117*	Hardened Steel	1/2" x 1"	2	0.2-0.6	1 oz
3118	Agate (SiO ₂)**	1/2" x 2"	2	0.2-0.6	1 oz
3127†	Hardened steel	3/4" x 2"	5	0.5-1.0	2 oz
5004†	WC**	3/4" x 2"	5	0.5-1.5	4 oz
8001	Hardened steel	2 1/4" x 3"	55	3-10	2 lb
8003	Alumina ceramic**	2 1/4" x 2 3/4"	60	5-10	1 lb
8004	WC**	2 1/4" x 2 1/2"	60	3-10	2 lb
8006	Methacrylate	2 1/4" x 2 3/4"	60	3-10	4 oz

(Includes 6 Lucite Center Sections)

*Multiple samples can be accommodated with 8010 Adapter in 8000 Mixer/Mill

†Multiple samples can be accommodated with 8011 Adapter in 8000 Mixer/Mill

**Because this material is brittle as well as hard, caution is required to prevent chipping and breaking, against which no guarantee can be offered; with the proper technique (instructions are included) hundreds of laboratories obtain years of grinding service.

GRINDING TESTS USING 8000 MIXER/MILL

Material	Form	Method	Time min	Amount grams	% Passing 325 mesh
Antimony	Pieces	L-D	5	26	97
Asbestos	Fluff	WC-D	10	*	*
Bauxite	60 mesh	TS-W	30	3	*
Bismuth	Chunks	PJ-D	20	5	75
Bone	Chunk	AC-D	10	*	55
Boron Carbide	Chunk	WC-D	15	7	100
Brake Linings	Chunk	WC-D	*	*	*
Carbon (activated)	Pieces	TS-D	10	10	90
Carnauba Wax	Piece	PJ-D	2	5	20
Cement (portland)	Powder	AC-W	30	20	100
Chrome Ore	Chunk	WC-D	10	15	39
Chromium	Chunk	WC-W	20	10	50
Cobalt		WC-W	10	10	91
Copper Shot		WC-D	15	2	95
Ferro Cr	100 mesh	WC-W	20	5	94
Ferro Nb		WC-W	60	5	10
Floor Tile	Chunk	WC-D	**	**	**
Germanium	Pieces	L-D	5	5	38
Ilmenite	Grains	WC-D	10	5	98
Limonite Ore	Grains	TS-W	30	3	100
Porcelain	Chunk	WC-D	15	6	83
Potassium Pyrosulfate	Fused/ Button	PV-D	10	5	100
Reforming Catalyst	Beads, 1/8"	AC-D	5	5	*
Sand	Grains	WC-D	2	12	86
Silica	Chips	L-D	30	15	*
Silica	Chips	AC-D	20	5	97
Silicon	Chunks	WC-D	15	10	92
Silicon	Lumps, 1/4"	L-D	10	5	30
Slag (blast furnace)		TS-W	20	3	100
Slag (copper)	100 mesh	WC-W	10	5	84
Slag (open hearth)		TS-W	20	3	100
Straw		TS-D	10	5	**
Ti-diborate		WC-D	15	5	100
Tomato Stems		TS-D	10	5	**
Transite	Chunks	WC-D	*	*	*
Tungsten Carbide		WC-W	15	10	100
Tungsten	Lumps	WC-D	10	25	50
Welding Flux		WC-W	30	5	82
Wood	Pieces	AC-D	10	1	50
Zirconium Carbide		AC-W	30	15	100

TS—No. 8001 Tool Steel Vial

AC—No. 8003 Ceramic Vial

WC—No. 8004 Tungsten Carbide Vial

PV—No. 6133 Polystyrene Vial

PJ—No. 8002 Polystyrene Jar

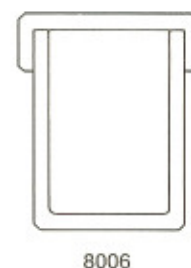
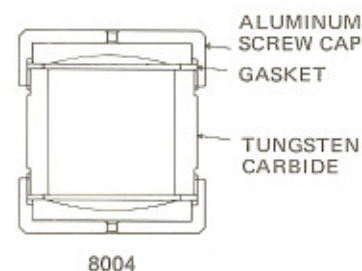
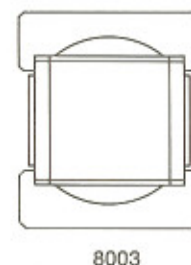
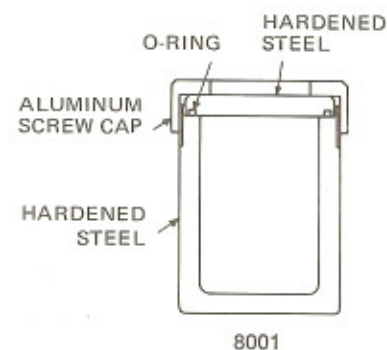
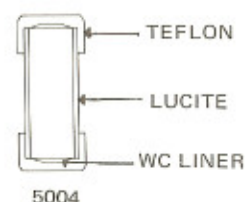
L—No. 8006 Lucite Vial

-D—Dry ground

-W—Wet ground (water or 1, 1, 1-trichloroethylene slurry)

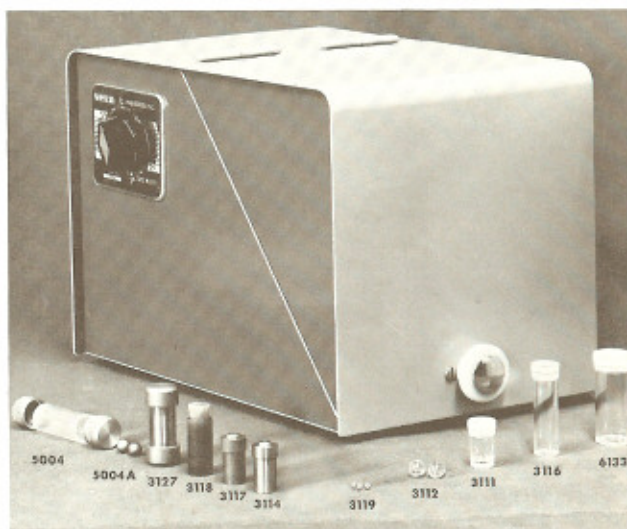
* Suitable for X-ray or Emission Spectroscopy

** Satisfactory for Extractions



No. 5100 Mixer/Mill®

Like the larger No. 8000 Mixer/Mill, the high grinding and mixing efficiency of the 5100 is a result of its three-dimensional action. Along the axis of the vial there is a component 1" long; at right angles 3/16" wide and up and down the oscillation is about ¼". The resulting action is consistent with the general shape and size of the vials. That is, at every stroke there is an impact at the end of the vial — over 100 per second — to crush the material rapidly and reproducibly. Among its advantages over other small mixers are:



- 1) The Mixer/Mill jaws are continuously adjustable accommodating any size vial from 1" to 2-¼" long and up to ¾" ϕ with no special adapters required.
- 2) Two vials may be shaken simultaneously.
- 3) It is extremely rugged with a strong enough motor to oscillate at 3200 rpm with a full load, assuring rapid grinding.
- 4) A unique timer switch runs the instrument for periods up to 6 minutes or, at another setting, up to one hour.
- 5) A protective and attractive housing encloses the entire mechanism including the vials during operation of the instrument.
- 6) Its motion along all three axes exceeds that of any other instrument its size affording more vigorous and therefore faster grinding action.

One to four samples will be ground at no charge, a full report and recommendations returned within two weeks. (See P. 15)

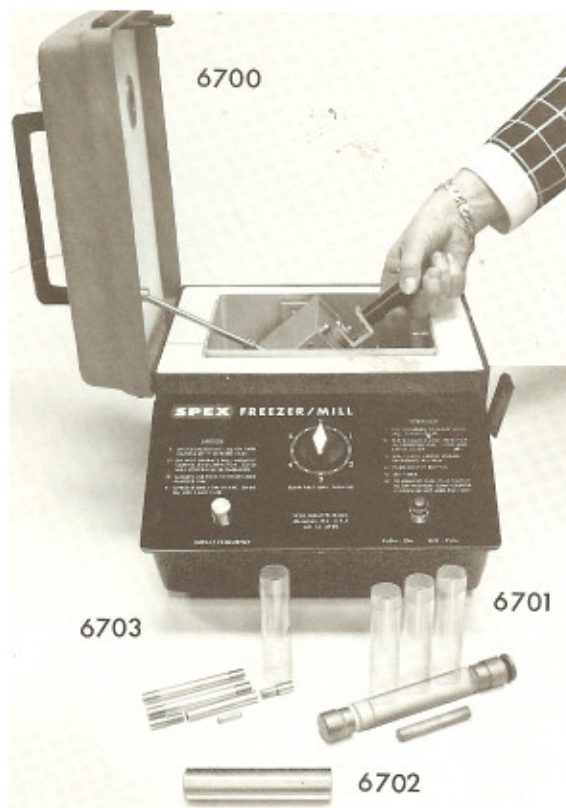
5100 Mixer/Mill®, 115V, 60Hz, or 230V, 50 Hz (specify), for quantities under 10 ml, dual timer permits mixing up to 6 or 60 min, built for heavy duty use with continuously variable jaws holding vials up to ϕ ¾" x 2¼" long, safety enclosure, 12" x 8" x 8", 22 lb

Grinding Containers

	Material	O.D.	Capacity, ml	Recommended load, ml	Weight
3111	Polystyrene	½" x 1"	2	1	4 lb/M
3116	Polystyrene	½" x 2"	5	3	7 lb/M
6133	Polystyrene	¾" x 2"	10	5	14 lb/M
3112	Methacrylate Ball	3/8" ϕ			
3119	Methacrylate Ball	1/8" ϕ			
3114	Stainless steel	½" x 1"	2	0.2-0.6	1 oz
3117	Hardened steel	½" x 1"	2	0.2-0.6	1 oz
3127	Hardened steel	¾" x 2"	5	0.5-1.0	2 oz
3118	Agate (SiO ₂)*	½" x 2"	2	0.2-0.6	1 oz
5004	WC*	¾" x 2"	5	0.5-1.5	4 oz

(Includes 6 Lucite Center Sections)

*Because this material is brittle as well as hard, caution is required to prevent chipping and breaking, against which no guarantee can be offered; with the proper technique (instructions are included) hundreds of laboratories obtain years of grinding service.



No. 6700 Freezer/Mill®

Reg. U.S. Pat. Off.

The Spex 5100 and 8000 MIXER/MILLS, operating at room temperature, readily grind most types of samples. Some types are so supple, however, that they bounce around unscathed in the vials. Under LN_2 nearly all material becomes friable. The FREEZER/MILL takes advantage of this property of materials by containing the sample in vials with magnetic end caps and rod-pestle but with non-magnetic center sections in a LN_2 bath. Thus, when the coils of the 6700 are alternately energized by the solid state circuitry the rod-pestle is driven back and forth between the end caps, at up to 30 times per second, grinding the sample. No local heating occurs; decomposition of temperature-sensitive compounds is averted.

One to four samples will be ground at no charge, a full report and recommendations returned within two weeks. (See P. 15)

6700 Freezer/Mill®, impact grinder for operation in self-contained liquid nitrogen bath, 115V, 50-60 Hz, includes 6704

6701 Grinding Vial, including two end plugs, an impactor and four plastic (polycarbonate) center sections

6702 Center Cylinder, stainless steel, for 6701 grinding vial when samples must be ground and remain free from organic contaminants

6703 Micro-Vial Set, stainless steel, 0.6 ml grinding capacity, set of three in special holder; for preparation of mixes of micro-samples with KBr

6704 Extractor & Vial Opener, for removing sample while frozen

6700 FREEZER/MILL GRINDING RESULTS

Material	Form	Weight, g	Time, min	Final Mesh
Nylon (a)	1/8" beads	2	2x2 (b)	100-200
Teflon	2 mil tape	3	2x2	100
Polyethylene	10 mil sheet	1	2	200
Candle wax	chunk	1.5	2	100-200
Chewing gum	chunk	1.5	2	100-200
Hair	dog clipping	0.5	2	200
Sheep wool	wad	0.5	2	200
Rubber, oil-extended	shearings 3/16"	1.5 (c)	2	25-50
Rubber band	shearings 3/16"	1.5	2x2	100
Space food	stick	2	2	100
Aluminum foil	2 mil piece	1 (d)	3x2	100-200
Steel wool	wad	0.5	2	100
Permalloy 5	shot 1/16"	2	3	30
Mouse skin	Raw, 1/2 animal	2 (e)	3	200

(a) Three different types of nylon yielded similar results.

(b) Two 2-minute grinds with a one-minute cooling period between.

(c) Equal amount of sand added. Purpose: ethanol-toluene extraction.

(d) 0.5g of Tide detergent added.

(e) Equal weight of sodium sulfate as dehydrating agent.

Wig-L-Bug

A widely accepted dental tool for triturating amalgams, the standard instrument is particularly useful for mixing and grinding small samples of less than 1 ml.

Use the plastic vials for mixing powders or preparing mulls with mineral oil. For preparing KBr pellets, use stainless steel vials, and grind for under 1 minute.



3110B Wig-L-Bug, black housing, 115V, 50-60 Hz, with 3113 vial adapter, 10" x 4" x 6", 10 lb

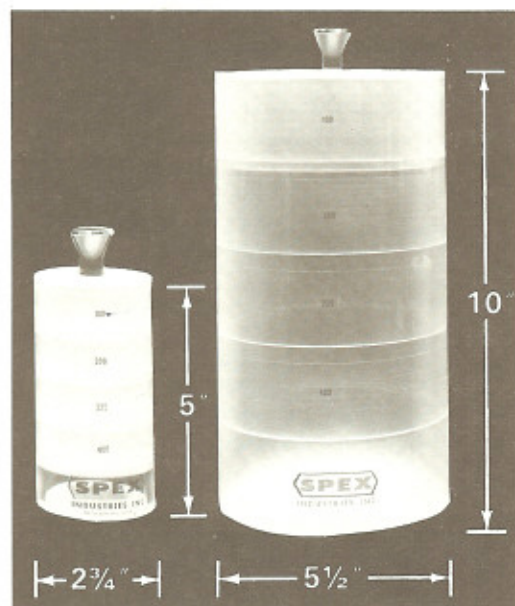
3110W Wig-L-Bug, ivory housing, 115V, 50-60 Hz, with 3113 vial adapter

3140 Wig-L-Bug, 115V, 50-60 Hz, with 3113 vial adapter, 1-hour timer and fan-cooled motor

3113 Adapter for $\phi \frac{1}{2}$ " x 1" long vials

3113K Adapter for preweighed KBr glass vial (see Spex chemical catalog)

Nylon Sieves



Every step in the handling of samples is a potential source of contamination. Our nylon sieves were designed to eliminate one such source of metallic impurities.

The four sieves, 100, 200, 325, and 400 mesh monofilament nylon cloth, are each stretched on an "embroidery" frame of telescoping methacrylate cylinders which stack over a receiving tray. The screens meet ASTM specification E11-58T for size and uniformity of mesh, can be quite easily replaced when worn or to avoid intercontamination of materials and are available in packages of pre-cut circles. The whole assembly slips apart for easy cleaning.

3536 Sieve Set, consisting of 4 frames and 1 tray with 1 each of the screens listed below

3530 Sieve Frame, consisting of two telescoping methacrylate rings 57 mm i.d. x 25 mm high, specify for 100, 200, 325 or 400 mesh

Screens, nylon monofilament cloth 88 mm ϕ

3531 100 mesh screen (12)

3533 325 mesh screen (12)

3532 200 mesh screen (12)

3534 400 mesh screen (12)

3535 Tray, 67.5 ml, 57 mm i.d. x 25 mm high

3546 Sieve Set, consisting of 4 frames and 1 tray with 1 each of the screens listed below

3540 Sieve Frame, consisting of two telescoping methacrylate rings 120 mm i.d. x 50 mm high, specify for 100, 200, 325 or 400 mesh

Screens, nylon monofilament cloth 150 mm ϕ

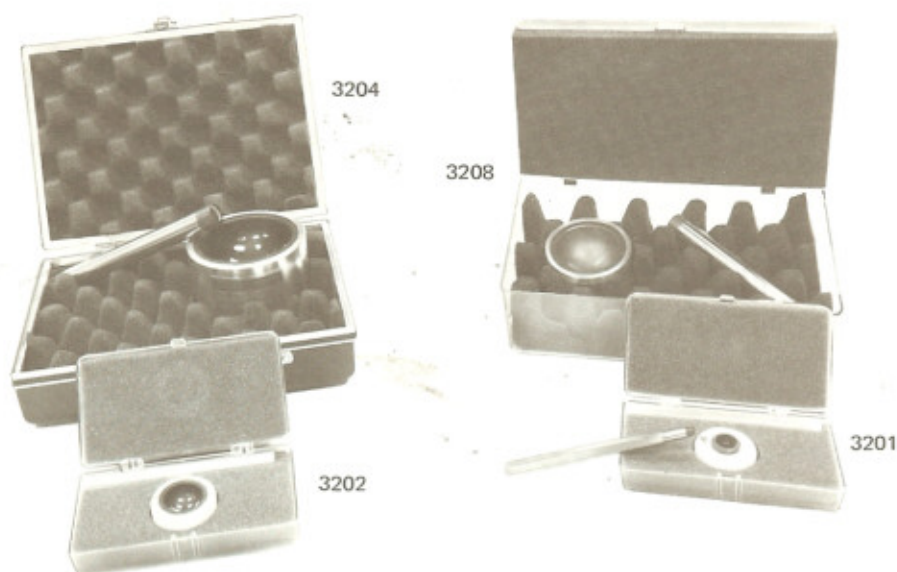
3541 100 mesh screen (12)

3543 325 mesh screen (12)

3542 200 mesh screen (12)

3544 400 mesh screen (6)

3545 tray, 540 ml, 120 mm i.d. x 50 mm high



BORON CARBIDE

Boron carbide is one of the best materials for hand grinding. Possessing a hardness close to diamond, it is also extremely inert, resisting attack by most acids and alkalis. In addition, boron carbide is unbonded so that the only possible metallic contaminating element is boron itself. Here it differs from, say, tungsten carbide which is usually bonded with cobalt. Following is a comparison with other materials as to Knoop hardness number:

MATERIAL	KNOOP HARDNESS
Tungsten Carbide	1050-1500
Aluminum Oxide	1265-1630
Silicon Carbide	2130-2140
Sapphire	1600-2200
Boron Carbide	2250-2260
Diamond	6000-6500

The ½" and 1" ϕ mortars are mounted in removable plastic bases for ease in handling; 2" ϕ and larger are encased in aluminum. Pestles are attached to an aluminum handle.

SILICON CARBIDE

In spite of the evidence above attesting to the superiority of BC as a grinding material, at the three inch diameter, economics becomes a significant factor and many labs have frugally been making do with not-so-hard, not-so-durable and not-so-expensive alumina mortars. A Silicon Carbide Mortar and Pestle Set costs less than half that of boron carbide. Again consulting Knoop we find silicon carbide hardness right up there, double that of tungsten carbide and within the range of sapphire.

Our 3208 is unpolished but smooth and, but for the extreme grinding problems still requiring boron carbide, will be just as efficient and comparably contaminant-free. Silicon carbide is inert to most solvents and contains no binder.

3201 Mortar and pestle, boron carbide. Mortar cavity ϕ ½" by 5/32" deep, highly polished. Pestle ϕ ¼"

3202 Mortar and pestle, boron carbide. Mortar cavity ϕ 1" by ¼" deep, highly polished. Pestle ϕ ½"

3205 Mortar and pestle, boron carbide. Mortar cavity ϕ 1½" by ¾" deep, highly polished. Pestle ϕ 9/16"

3203 Mortar and pestle, boron carbide. Mortar cavity ϕ 2" by 1" deep, highly polished. Pestle ϕ 9/16"

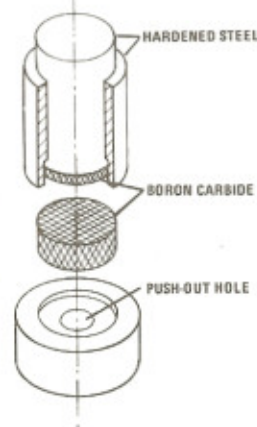
3204 Mortar and pestle, boron carbide. Mortar cavity ϕ 3" by 1½" deep, highly polished. Pestle ϕ ¾"

3208 Mortar and pestle, silicon carbide. Mortar cavity ϕ 3" by 1½" deep; pestle ϕ ¾"

Mortars and Pestles

Boron Carbide Crusher

For rapidly crushing rocks and hard, brittle synthetics or minerals this modern version of the old Plattner's Diamond Mortar is ideal. The sample, in the form of chunks up to about 8-mm ϕ , is sandwiched between two boron carbide discs. The top of the rod holding the upper boron carbide disc is struck a few times with a hammer. In an "acid test" an alumina ceramic ball succumbed, powdering within ½ dozen blows. A hardened steel compartment contains the powder; the only contaminant likely, and this in the ppm range or below, is boron carbide.



3210 BORON CARBIDE CRUSHER, 1-¼" ϕ discs supported in a hardened steel container

X-Ray Accessories

Claisse Fluxer

7153 Claisse Fluxer, for fusing up to six samples simultaneously; with 6 burners and supports for platinum crucibles; separate shaker control unit; 115V, 50/60 Hz or 230V, 50/60 Hz (specify); operates on natural, LPG, or artificial gas plus compressed air; requires platinum crucibles and lids

7154 Claisse Fluxer, as above but for use with LPG only; does not require compressed air

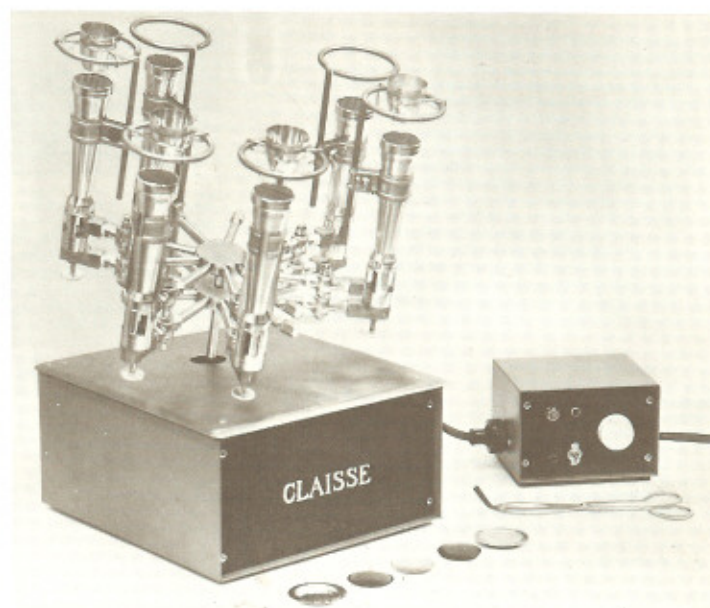
Note: Because of the special and expensive nature of platinum ware, the crucibles and lids needed for this apparatus are being supplied by the manufacturer (Engelhard Industries) rather than ourselves. Contact us, however, if you need further information.



7151 Rack and Handling Tongs for 6 No. 7152 graphite crucibles, heli-arc welded high-temperature wire; tongs for placement in and removal of rack from furnace . . .

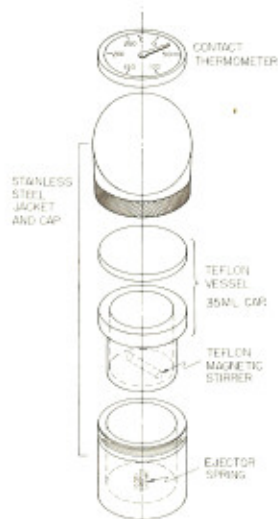
7152 Crucible, graphite, (regular grade, not high purity), ϕ 1 1/4" x 1" long, 9 ml capacity.

6005 Lithium Tetraborate



Prof. Fernand Claisse of Laval University in Quebec who in 1956 (Quebec Dept. Natural Resources P.R. 327) introduced the well accepted fusion technique for homogenizing samples prior to x-ray spectrometric analysis, has now greatly improved the productivity of the method with a semi-automatic fluxing apparatus. Mixed together with lithium tetraborate or another suitable flux, up to six samples may be loaded into non-wettable PtAu crucibles. These are mounted on a special, variable-speed shaker above individual gas burners. Heated to a temperature above 1000C, sample and flux fuse intimately and uniformly within a few minutes. The lid of the crucible serves a dual purpose: First, it restrains the molten material from sloshing out; second, it becomes the casting mold. Crucible and lid are simply turned upside down to cast a perfect disc. It is of the right diameter to fit standard x-ray holders; it is bubble-free, homogeneous and contains a built-in internal standard if desired; most important, its bottom surface is flat to assure reliable x-ray data.

Decomposition Chamber



Most silicate minerals, ores, slags, glasses, Portland cements, and ferro-alloys dissolve rapidly when digested in an appropriate mineral acid at elevated temperatures and pressures. The new Spex Decomposition Chamber accomplishes this without contamination while retaining volatile components. And safely. Reactants contact only a removable Teflon vessel and its tightly clamped Teflon lid. Should internal pressure exceed a safe value, the Teflon lid will rupture relieving the pressure through a small hole bored in the outer stainless steel cap.

Constructed entirely of 18-8 stainless steel and Teflon, the Decomposition Chamber will resist corrosion to remain labworthy for years to come. Heating up to 150C can be conducted either in an oven or on a hotplate. Typically, 200 mg of a ground silicate mineral are dissolved in 5 ml of HF when kept in the Chamber at a temperature of 115C for 15 minutes. The entire assembly may be immersed in water to cool it before the knurled cap is unscrewed.

A disc-type surface thermometer resting on top of the tilted lid allows convenient monitoring of the digestion; a Teflon-covered rod is supplied for magnetic stirring. You will want more than one of these Decomposition Chambers for your x-ray, atomic absorption, optical emission or wet chemical analyses.

7155 Decomposition Chamber, Teflon vessel with Teflon lid inside a screw-capped stainless steel container

B-25 Press

This press was developed particularly for analysts and spectroscopists. Its design is based on a double-ring frame which provides unusual access to the work area from all four sides. The upstroking 3-5/8" diameter ram has a movement of one inch (25 mm) and compresses the work against an upper screw which is fitted with a cranked handle for easy height adjustment. A pressure release valve helps prevent damage to specimens by allowing the maximum pressure to be regulated from zero to 25 tons.

The B-25 press features:

Compactness, Continuous and quick adjustment between ram and upper screw, Accessibility from all four sides, Adjustable pressure relief valve, Extremely light weight, Economical price.

Total Pressure	25 Tons	25,000 Kg
Platen Movement	1 inch	2.5 cm
Lower Platen Diameter	3 1/4 inch	8.2 cm
Maximum Daylight	4-7/8 inch	12 cm
Minimum Daylight	1 1/2 inch	3.8 cm
Upper Screw Adjustment	2-3/8 inch	6 cm
Horizontal Access	360° arc	
Height	23 inch	58.4 cm
Depth (base)	8 inch	20 cm
Width (base)	11 1/4 inch	29.5 cm
Net Weight	112 lb	50 Kg
Crated Weight	~150 lb	~67 Kg

Spec-Caps



Spec-caps eliminate the need for backing materials or binders when pressing 1 1/4" pellets.* Reinforced by the thin-walled aluminum cups, briquets are safely and easily handled without risk of breaking and losing time-invested samples. The painted outside surface prevents mold sticking and permits marking for identification and storage of the pellets as standards.

*C.K. Matocha, App. Spec., 20, 252, 1966

3619 Spec-cap, ϕ 1.185" x 0.325" thick, produces briquets ϕ 1.235" x 3/16" thick, requires 3623 die

X-Ray Cells

Nearly every petroleum refinery in the country relies on these polypropylene cells in preparing heating oil samples for x-ray sulfur determinations.

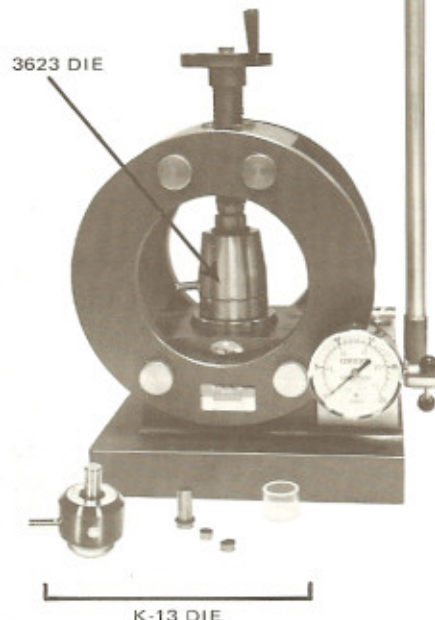
They have also been adapted by ingenious customers for solution residues and powders. If, after preparing the Mylar window normally, it is dished slightly with a round-ended glass rod a drop of solution, placed there and warmed under an infrared lamp, will be dried to a smudge that can be analyzed in situ. If, instead, powder is spread on the taut Mylar surface and another piece of Mylar overlaid with a second ring, the sandwich filling is ready for x-ray analysis.

Volatile samples such as light petroleum fractions can be analyzed by venting the cell with a small pinhole and leaving a little air room for expansion.

3513 Placement Tool for slipping O-ring over x-ray cell
3515 Expendable x-ray cells, polypropylene

3517 Mylar, 1/4-mil film, 2 1/2" x 300 ft

3518 Aluminum Spacer, specify 1/4", 1-3/8", 2" to fit sample cavity of spectrometer
3519 Expendable O-Rings for #3515 cells



B-25 Hydraulic Press, 25 ton

K-13 Evacuatable Die, may be used either with or without vacuum; optically flat, polished and parallel hardened tool steel faces; produces pellets ϕ 13 mm x up to 1/4" thick, 3 lb

3623 Evacuatable Die, may be used either with or without vacuum; optically flat, polished and parallel hardened tool steel faces; produces pellets ϕ 1.235" x up to 5/16" thick. Recommended for use with vacuum x-ray spectrometers, 10 lb

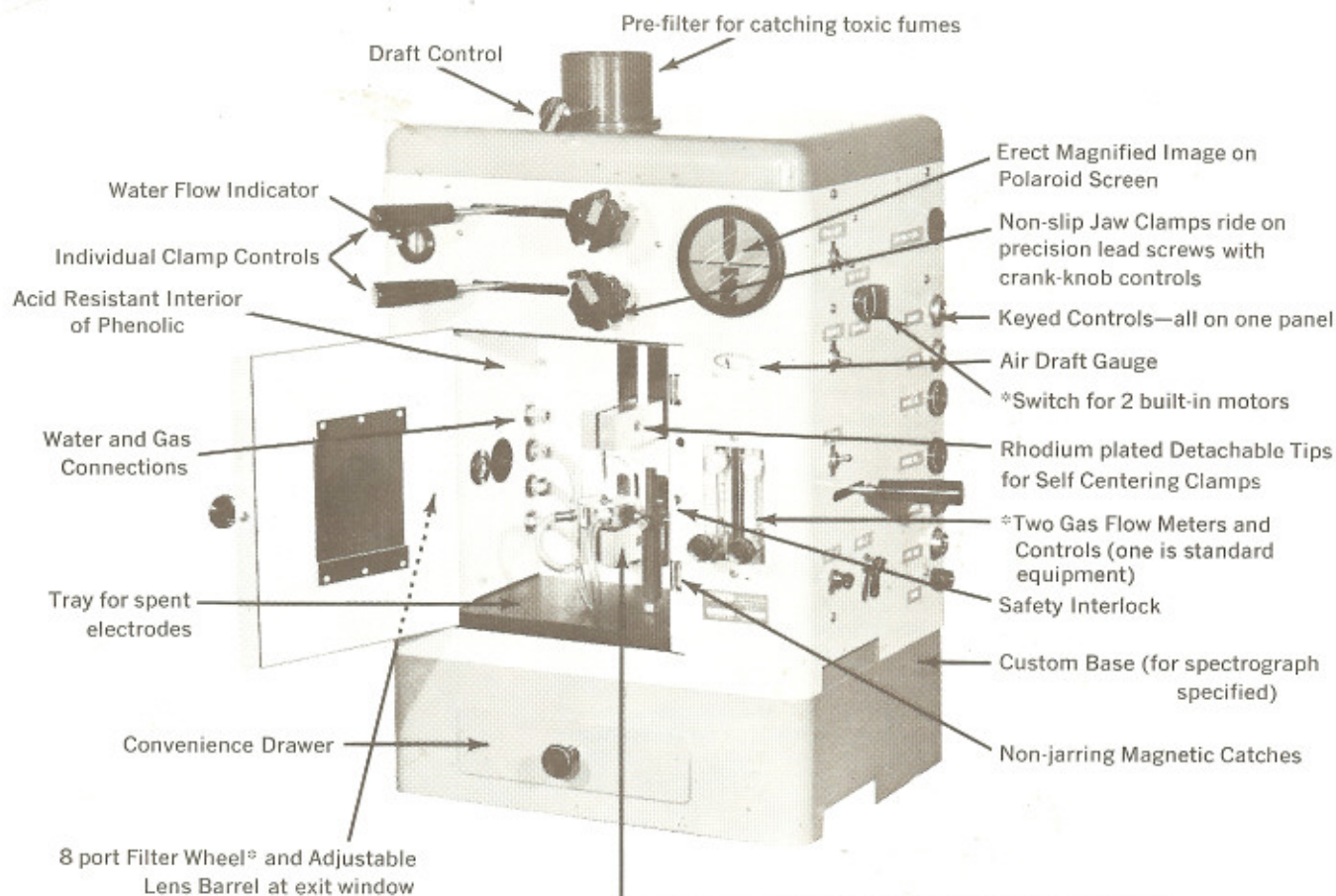
3623C* Tungsten Carbide Pellets, ϕ 1.235" for 3623 die

*Because this material is brittle as well as hard, caution is required to prevent chipping and breaking, against which no guarantee can be offered, with the proper technique (instructions are included) hundreds of laboratories obtain years of grinding service.



Arc/Spark Stand

Left or right hand models adapt to most new spectrographs or will modernize your existing equipment.



*OPTIONAL ACCESSORIES

SNAP-IN ACCESSORIES INCLUDE:
 *Stallwood Jet (shown) for cleaning up background and stabilizing arc
 *Plasma Jet for trace analysis of liquids
 *Petrey Stand for point-to-plane work
 *Atmosphere Chamber for Petrey Stand
 *Combination Analyzer (Rotrode and Platrode)

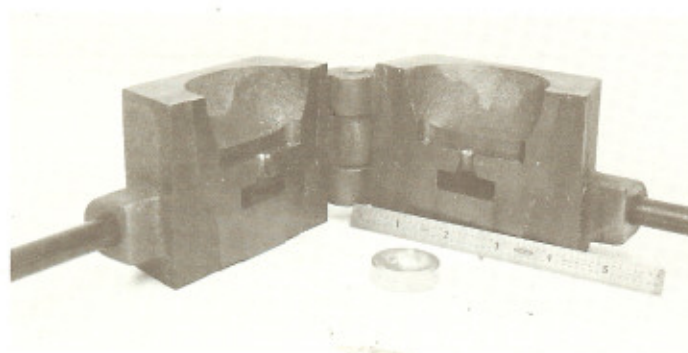
Ask for separate detailed Arc/Spark Stand catalog.

We also carry a complete line of graphite products, photographic supplies and standard materials. Send for applicable catalog.

9010 Arc/Spark Stand, 115V, 50-60 Hz, includes Flowmeter and Water Cooling Connections, specify type of spectrograph and the direction light must emerge as operator views stand, 15" x 12" x 21", 129 lb

9010A Adapter For B&L Spectrograph

All Others



This cast iron book mold for preparing discs of low-melting alloys ϕ 1 1/4" by 3/8" thick permits spectrographers to match their samples to standard discs and so facilitates set-ups and increases accuracy.

The large mass of the mold quickly freezes the casting and the sample can be removed almost immediately. The disc is cast horizontally, promoting fast chilling and resulting in small, uniform grain structure on the surface of the sample to be analyzed. A minimum of machining or sanding is required because the faces of the mold are machined quite smooth.

1 1/4" Disc Book Mold

3904 Book Mold for casting discs about ϕ 1 1/4" x 3/8" thick, 16" x 3" x 3", 12 lb

Questionnaire for Test Grinding of Samples

(One to four samples will be ground at no charge, a full report and recommendations returned within two weeks.)

Composition of sample _____ Suggested Spex Grinder _____

Quantity to be ground (or mixed) _____
in a single load _____

Purpose of grinding:

Emission spectrographic analysis _____ extraction _____

x-ray spectrographic analysis _____ other _____

Number of samples to be ground per day _____

Is contamination at around 0.1% from any of the following objectionable? Specify which.

Iron _____ Tungsten Carbide _____

Alumina _____ Cobalt _____

Acrylic _____ Polystyrene _____

Ultimate particle size desired _____

_____ Company

_____ Representative
(name and position)

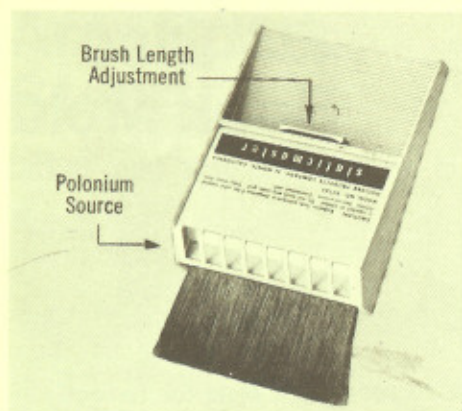
_____ Mailing address for sample
return and report

DATE: _____

SPEX INDUSTRIES, INC. • BOX 798, METUCHEN, N.J. 08840 • (201)-549-7144

WESTERN REGIONAL OFFICE: 129 ARCADIA AVE., SANTA CLARA, CALIF. 95051 (408)246-2333

Staticmaster Brushes



3901 Staticmaster Brush, 3" wide. Model 3T500 (500 microcurie polonium element)

3902 Staticmaster Brush, 1" wide. Model 1C200 (200 microcurie polonium element)

Ordering Information

Terms are net 30 days to rated firms. To avoid delays purchasers who have not transacted any previous business with Spex Industries should include commercial references or remittance with the initial order.

Shipments of orders over \$25.00 will be made F.O.B. Destination anywhere within the continental United States via our choice of surface transportation. Any premium transportation, packaging or invoicing requested will be additionally invoiced. A \$2.00 minimum postage and handling charge is made for orders under \$25.00. A minimum order of \$10.00 is required.

Guarantee

Our products are guaranteed:

- (1) to conform to the specifications of the most recent model of the item.
- (2) against defects of workmanship and parts for one year from the date of the original shipment.

Although catalog information is as representative of the product as possible, we must reserve the right to make changes in specifications or prices, and also to delete and add items.

We would be pleased to update your files on equipment catalogued elsewhere.
Our products include:

Spectrochemical Standards

Mass and Thermal Chromatographs

Laser-Raman Spectrophotometers

High Purity Materials

Single and Double Monochromators,
Spectrometers, Spectrographs from
0.1 through 1-meter fl

Electrodes, Plates and Film
for Emission Spectroscopy

WESTERN REGIONAL OFFICE: 129 ARCADIA AV., SANTA CLARA, CA. 95051 408/246-2333

SPEX

INDUSTRIES, INC./P.O. BOX 798/METUCHEN, N. J. 08840