14th Edition SPEX® SamplePrep

HANDBOOK

OF SAMPLE PREPARATION & HANDLING

Grinding & Pulverizing
Tissue Homogenization & Cell Lysis
Mixing & Blending
Electric Borate Fusion
Pressing & Pelletizing

SPEX® SamplePrep®

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A SPEX® SamplePrep Company

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SPEX SamplePrep, LLC has been providing superior sample preparation equipment and supplies since 1954. Our mission is to provide quality products backed by exceptional service and expertise. Our equipment is used to prepare samples for a wide range of analytical technologies including XRF, ICP, GC-MS and PCR. Much of our equipment is also used for cutting-edge research in pharmaceuticals, superconductors, polymers, forensics, genetics and biotechnology.

CORPORATE LOCATIONS

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Vertically moving clamp.

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OUR PRODUCTS

Freezer/Mill®
Uniquely designed to cryogenically grind samples normally considered difficult or impossible to pulverize at ambient temperature.

Geno/Grinder®, MiniG® & GenoLyte®
Used for high-throughput cell disruption and tissue homogenization.

Mixer/Mill®
High-energy ball mills for pulverizing brittle materials, mixing powders and emulsions, mechanical alloying, and nanomilling.

Katanax® Electric Fusion Fluxers
Automated electric fluxers for borate fusions of cement, ores, ceramics and other materials for analysis by XRF, AA & ICP.

ShatterBox®
Ring and puck mill used for rapidly pulverizing brittle materials such as cement, slag, ores, ceramics, etc.

XRF Accessories
Spec-Caps® for pressed disks, disposable X-CELLS® and window film for liquid samples, Prep-Aid® Binders and Grinding Aids.

X-Press® & Manual Presses
Automated and manual laboratory presses ideal for pressing sample disks for XRF, IR and OES.

OUR CUSTOMERS

For the past 65 years, SPEX SamplePrep's customers have relied on our unique knowledge and expertise to select the proper products for their sample preparation needs. Our exceptional sales and service teams recommend and support reliable sample preparation equipment for the following fields:

- Cement Manufacturing
- Mining
- Geology & Mineralogy
- Polymers & Plastic
- RoHS/WEEE
- Medicine & Genetics
- Pharmaceuticals & Drug Testing
- Environmental Protection & Remediation
- Cosmetics
- Food & Agriculture
- Forensics & Crime Scene Investigation
- Materials research

SAMPLE PREPARATION TECHNIQUES

Sample preparation techniques are the building blocks used to prepare samples for specific scientific analyses. They are the foundation for ensuring high quality and reliable analytical results. Our equipment and accessories are used for a variety of common techniques as well as specialities, such as cryogenic grinding. Listed below are the symbols we use to identify each technique and some insight into the use of our equipment.

GRINDING & PULVERIZING

Our laboratory mills and grinders are capable of pulverizing samples in both ambient and cryogenic environments. This versatility allows for grinding of a wide variety of samples, including metals, rocks, plastics, plant and animal tissue, bone and pharmaceuticals.

MIXING & BLENDING

The nonhomogeneity of materials in the real world presents a serious sampling problem for scientists. Our mills are effective at mixing and blending dry materials such as pigments, powders, pharmaceuticals and cosmetics as well as samples that require mixing for effective extraction with a solvent or buffer.

TISSUE HOMOGENIZATION /CELL LYSIS

Effective cell disruption and DNA/RNA extraction are critical for biotechnology and forensic applications. Our Geno/Grinders and Freezer/Mills aid scientists by providing high-throughput sample preparation solutions at both ambient and cryogenic temperatures.

PRESSING & PELLETIZING

Analytical spectroscopic methods such as XRF, OES, and IR often require samples in the form of a round, flat-surfaced disc. Our laboratory presses are ideal for preparing sample discs for these methods. In addition, our automatic and manual presses are designed to fit on the bench top, requiring less space than standard floor model presses.

BORATE FUSION

For samples that are difficult to prepare as homogeneous pressed powders (cement), difficult to dissolve in acid (zirconia and alumina), or both (metal ores and silicate rocks), borate fusions are widely used. SPEX SamplePrep offers the full line of Katanax Electric Fusion Fluxers & Fusion Fluxes for preparation of samples for XRF, AA, and ICP.

CONTENTS

TISSUE HOMOGENIZERS

Our range of tissue homogenizers are designed with a unique, vigorous pulverizing motion and an adjustable clamp that handles various sizes of titer plates and vials. They are specifically designed for rapid cell disruption, cell lysis, and homogenization of plant and animal tissue through bead beating. This enables fast and efficient extraction of nucleic acids, DNA, proteins and other molecules. A full range of Kryotech® accessories are available to preserve temperature sensitive samples such as proteins and RNA. Our homogenizers are also ideal for the QuEChERS method used for extracting pesticide residues and other organic compounds.

Our homogenizers enable increased extraction efficiency and reproducibility over traditional sample preparation methods. Sample preparation is usually complete in two minutes or less. Samples are processed in sealed vials or plates, so no cleanup is required and cross contamination is minimized.

The **2010 Geno/Grinder®** is an automated high-throughput homogenizer that accommodates a full range of sample vials and deep-well titer plates.

The **1600 MiniG®** is a smaller homogenizer for labs that process fewer samples. It accommodates a full range of sample vials or deep-well titer plates.

The **1200 GenoLyte®** is a compact homogenizer that processes samples in a range of vials.

The **2030 Geno/Grinder®** is a fully automated homogenizer that integrates with most automated robotic systems and platforms.

APPLICATIONS	SAMPLE TYPES
DNA/RNA extraction	Animal & Plant tissue
Cell Lysis	Cell cultures
Pesticide & other residue extraction	Fruit/Vegetables
QuEChERS	Cannabis
Protein extraction	Seeds
Biofuels	Yeast
Food Safety	Bacteria
	Cereals

2010 GENO/GRINDER®

The 2010 Geno/Grinder® is a high-throughput homogenizer with an adjustable clamp that accommodates a full range of sample vials from 2 to 50 mL, jars up to 750 mL, or up to six deep-well titer plates. It is specifically designed for rapid cell disruption, lysis and tissue homogenization. Kryotech® accessories are available to preserve temperature sensitive samples during the grinding process.

Window in lid allows you to see the grinding process as it happens.

Safety lock prevents lid from being opened during grinding process.

Ambient status light.

Vertically moving clamp ensures consistant sample processing.

Clamp has quick-release button to adjust for vials or titer plates.

LCD control panel run times in minutes: seconds format.

Rate/timer controls and start/stop buttons.

TOUCH SCREEN CONTROL PANEL

The fully programmable control panel can be tilted to give the user an optimal viewing angle. Programmable parameters include run time, rate, cycles, and pause time. Administrative lockout features (password protected) allows lab manager to restrict user access to specific protocols. Run history is recorded and can be exported via USB. The Geno/Grinder records information for service.

CONTROL PANEL

- Time can increase and decrease by 5 second increments
- Rate can be adjusted by increments of 5

SAVED PROTOCOLS

Up to 500 protocols can be saved for fast, simple recall, increasing productivity and reducing operator error.

RESOURCES

Resources screen includes training videos and instructions, operating manual, a catalog of accessories and tech support information.

SPECIFICATIONS

VOLTAGE	100–120 V, 60 / 50 Hz 200–240 V, 50 / 60 Hz
CLAMP MOVEMENT	1.25 in (3.2 cm) vertical
MOTOR	1/3 hp
WEIGHT (LBS)	106 lbs. (48 kg)
TIMER/ CONTROLS	Digital display in minutes: seconds (max 20:00)
CLAMP SPEED	Adjustable from 500 to 1,750 strokes/ minute
POWER CORD	Supplied with a 3-prong grounded cord 115 V, 60 Hz, or a 2-prong grounded European cord for 230 V, 50 Hz.

MIXING & GRINDING OPTIONS

- Up to 96 x 2 mL or 5 mL vials
- 24 x 15 mL vials
- 16 x 50 mL vials
- 2 x 750 mL jars
- Jars up to 750 mL

- Up to 6 deep well titer plates.

- Cryoblocks available for all vials.

1600 MINIG®

The 1600 MiniG® is ideal for labs that need a small yet powerful tissue homogenizer. It has an adjustable clamp that accomodates samples vials or titer plates. Specifically designed for rapid cell disruption, lysis and tissue homogenization. Kryotech® accessories are available to preserve temperature sensitive samples during the grinding process.

Window in lid allows you to see the grinding process as it happens.

Safety interlock switch stops motor if lid is opened during grinding process.

Start/stop buttons.

Rate and timer controls.

Vertically moving clamp ensures consistant sample processing.

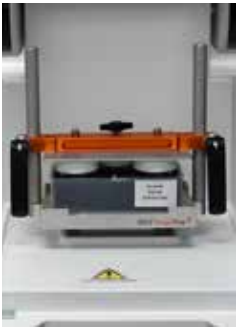
Adjustable clamp allows stacking to hold up to two layers of vials or titer plates.

LCD display screen shows run times in minute:second format.

SPECIFICATIONS

VOLTAGE	100–240 V, 60 / 50 Hz
CLAMP MOVEMENT	1.25 in. (3.2 cm) vertical
MOTOR	1/7 HP
WEIGHT (LBS)	44 lbs. (20 kg)
TIMER/ CONTROLS	Digital display in minutes: seconds (max 10:00)
CLAMP SPEED	Adjustable from 500 to 1,500 strokes/ minute
POWER CORD	Supplied with a 3-prong grounded cord 115 V, 60 Hz, or a 2-prong grounded European cord for 230 V, 50 Hz.

MIXING & GRINDING OPTIONS



- Holds up to 48 x 2 mL or 5 mL vials
- 12 x 15 mL vials
- 6 x 50 mL vials
- 2 x 75 mL jars

- Up to 2 deep well titer plates

- Cryoblocks avilable for all vials.

1200 GENOLYTE®

The 1200 GenoLyte® is the ideal solution for labs that need a compact yet powerful tissue homogenizer and cell lyser. It is equipped with interchangeable sample vial holders allowing a variety of vial types from 2 to 12 mL. It is designed for rapid cell disruption, lysis and tissue homogenization enabling fast and efficient extraction of nucleic acids, proteins and other molecules of interest. The powerful grinding motion of the GenoLyte is also able to grind hard materials such as rocks and minerals.

Digital display screen shows run times in minute : second format.

Start, stop and pause button.

Rate & timer controls.

Safety interlock switch stops motor if lid is opened during grinding process.

Clear lid allows you to see the grinding process.

Interchangeable vial holders for different vials sizes and types. (Sold separately.)

SPECIFICATIONS

VOLTAGE	100–240 V, 60 / 50 Hz
DIMENSIONS	15 in. (38 cm) long x 8 in. (20.5 cm) wide x 11 in. (30 cm) high.
MOTOR	1/7 HP
WEIGHT (LBS)	24 lbs. (11 kg)
CLAMP SPEED	750, 2000, 3000, 4000 RPM
POWER CORD	Supplied with a 3-prong grounded cord 115 V, 60 Hz, or a 2-prong grounded European cord for 230 V, 50 Hz.

MIXING & GRINDING OPTIONS

- 6 x 2 mL vials
- 4 x 5 mL vials
- 2 x 7 mL or 12 mL vials
- 1 x stainless steel or tungsten carbide 5 mL vial
- 1 x 3.5 mL agate vial

2030 GENO/GRINDER®

The 2030 Geno/Grinder® is an automated plant and tissue homogenizer that integrates with most robotic systems and platforms. Its vertical grinding motion prepares plant and animal tissue for extractions of DNA. The average processing time for each cycle is two minutes or less. It has a versatile clamp that accommodates most standard titer plates and vials.

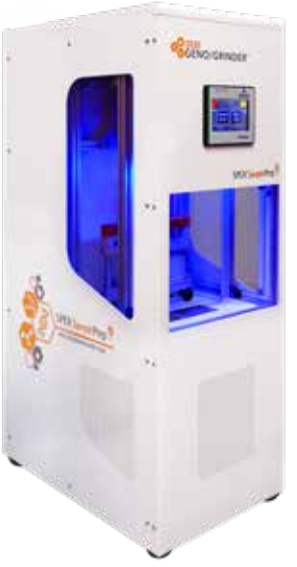
Clamp is automatically secured before the grinding process and accomodates most standard titer plates and vials.

Unique vertical grinding motion that consistently processes samples.

CE

SPECIFICATIONS

VOLTAGE	100–240 V, 60 / 50 Hz
DIMENSIONS	12.9 in. (32.7 cm) x 18.9 in. (48.0 cm) x 35.9 in. (91.0 cm)
CLAMP MOVEMENT	1.25 in. (3.2 cm) vertical
MOTOR	3-phase servo motors
WEIGHT (LBS)	129 lbs (58.5 kg)
CLAMP SPEED	500-1,750 strokes per minute
POWER CORD	Supplied with a 3-prong grounded cord 115 V, 60 Hz, or a 2-prong grounded European cord for 230 V, 50 Hz.



Auto Geno/Grinder® integrated with the Thermo Scientific VALet™

2011 ADJUSTABLE CLAMP FOR 2010 GENO/GRINDER®

The 2010 Geno/Grinder is supplied with the 2011 Adjustable Clamp. It holds up to three layers of deep-well titer plates and other titer plates, tubes and vials of equal or lesser height. The clamp has a hand-knob with a quick-release button to move it up or down a central threaded rod.

PLATES & TRAYS

2189T NESTING TRAY SET GENO/GRINDER®

Nesting trays used to stack titer plates vertically in the 2011 adjustable clamp. Set comes with two nesting mid-plates and one bottom plate.

1690T NESTING TRAY FOR MiniG®

Nesting trays used to stack titer plates vertically in the MiniG 1690 adjustable clamp.

GRINDING BALLS, DISPENSERS AND EXTRACTORS

Stainless steel balls are useful for grinding large or tough samples. The choice of ball size should be determined based on the sample material and the size of the grinding vial or titer plate. After use, the grinding balls can be discarded or cleaned for re-use.

2150 GRINDING BALLS, 5/32 IN. (4 MM)

Made of 440C stainless steel. Used with 2100 grinding Ball Dispenser and all 96-well titer plates. Sold in bags of 5,000.

2151 GRINDING BALLS, 4/32 IN. (3 MM)

Made of 440C stainless steel. For use in 2230 titer plate with 3116PC, 2241-PC and 2241-PEF vials. Sold in bags of 100.

2155 GRINDING BALLS, 3/8 IN. (9.5 MM)

Made of 440C stainless steel. For use in 2230 titer plate with 3116PC, 2241-PC and 2241-PEF vials. Sold in bags of 100.

2156 GRINDING BALLS, 7/16 IN. (11 MM)

Made of 440C stainless steel. For grinding large or tough samples in 2230 titer plates and 2250 vial sets. Sold in bags of 100.

2157 GRINDING BALLS, 1/2 IN. (12.7 MM)

Made of 440C stainless steel. For grinding large or tough samples in 2230 titer plates and 2250 vial sets. Sold in bags of 100.

2186 ZIRCONIA GRINDING BALLS

6 mm zirconia grinding beads. Sold in bags of 1,000.

2100 GRINDING BALL DISPENSER

Simultaneously dispenses one 5/32 in. (4 mm) steel grinding ball 2150 into each well of a standard 96-well titer plate, 2200 and 2210 titer plates.

2110M-12 MAGNETIC TIPS

Converts micropipette dispensers into pin magnets for removing grinding balls from deep-well titer plates. Sold in packs of 12 or 96 2110M-96.

GRINDING BEADS

Molecular Biology Grade Grinding Beads are treated to inactivate contaminating enzymes and have been tested accordingly. Low Binding Grinding Beads are coated to reduce non-specific binding of nucleic acids and proteins and are used for lysing dilute samples of cells. Acid Washed Grinding Beads are treated to remove fine particles and contaminants. Contact us for detailed instructions.

SILICA GRINDING BEADS

Acid washed silica grinding beads. 200 gram bottle. Available in bead sizes 800-1000 µm 2160 and 400-660 µm 2165.

LOW BINDING SILICA BEADS

Acid washed and chemically treated to keep samples from binding to titer plate wells. Available in sizes 800 µm 2162, 400 µm 2167, 100 µm 2168.

2166 SILICA GRINDING BEADSMOLECULAR BIOLOGY GRADE, 400-600 µm

Acid washed RNase/DNase-free treated silica beads. 200 gram bottle.

2180 ZIRCONIA GRINDING BEADS, MOLECULAR BIOLOGY GRADE, 200-400 µm

Acid washed RNase/DNase-free treated zirconia beads. 250 gram bottle.

2181 LOW BINDING ZIRCONIA BEADS

Acid washed and chemically treated to keep samples from binding to titer plate wells. 250 gram bottle. Availaible in 100 µm 2181 and 200 µm 2182.

2302-30 ZIRCONIUM BEADS, 3 MM

Acid washed zirconium beads, 300 g bottle.

GRINDING CYLINDERS

Our chemically inert ceramic cylinders are specifically designed for the QuEChERS method which is used to extract pesticide residues or other organic contaminants from fruits, vegetables, meat or seafood. The angle-cut ends help the cylinders to shear the sample matrix and break up salt agglomerates during processing, reducing extraction time and resulting in a thorough extraction of analytes into the solvent. Typically, 2 cylinders are used per sample vial.

CERAMIC GRINDING CYLINDER

Ceramic grinding cylinder with angle-cut ends. Sold in bags of 100. Available in large 3/8 in. x 7/8 in. 2183 for use with standard 50 mL centrifuge tubes, medium 5/16 in. x 5/8 in. 2184 for use with standard 15 mL centrifuge tubes and small 5/32 in x 5/16 in. 2185 for use with standard 5 or 15 mL centrifuge tubes.

TITER PLATES AND CAP-MATS

While most titer plates can be used in the Geno/Grinder, we carry sturdy titer plates that have been tested extensively with various samples and resist perforation by steel grinding balls even at high clamp speeds. They can be used for many applications including sample libraries, mother-to-daughter automated plate pipetting, large sample dilutions and cell suspensions. Suitable for RNA extraction when used with 2600 Cryo-Station. Titer plates are made of polypropylene, with alphanumeric marks for well identification. Cap-Mats seal titer plate wells, preventing spills and well-to-well contamination. Rugged, silicone rubber Cap-Mats may be sterilized and re-used.

2200–100 96-WELL TITER PLATE, SQUARE WELLS

Square 2.4 mL wells with a working capacity of 2.0 mL. Sold as a case of 100 titer plates or single 2200.

2201–10 CAP-MAT

For 2200 titer plates. Sealing mats for the 2200 titer plates, above. Sold in packs of 10 Cap-Mats or single 2201.

2210–100 96-WELL TITER PLATE, ROUND WELLS

Round 1.0 mL wells, rugged polypropylene, alphanumeric marks for well identification. Sold in packs of 100 or single 2210.

2211–10 CAP-MAT

For 2210 titer plate. Sealing mats for 2210 Titer Plates. Tough silicone rubber, prevents leaks and cross-well contamination. Sold in packs of 10 or single 2211.

2220–100 48-WELL TITER PLATE

For 2221 Cap-Mat. Rectangular 5 mL wells with a working capacity of 2 mL per well. Use with steel grinding balls 2150. Sold as case of 100 titer plates or single 2220.

2221–10 CAP-MAT

For 2220 titer plate. Cap-Mats seal 2220 48-Well Titer Plates. Sold as case of 10 Cap-Mats or single 2221.

2230–100 24-WELL TITER PLATE

Square 10 mL wells with a working capacity of 4 mL per well. Use with 2231 Cap-Mat, and one 3/8 in. (9.5 mm) steel grinding ball 2155. Sold as case of 100 or single 2230.

2231–10 CAP-MAT

For 2230 titer plate. Cap-Mats seal 2230 24-Well Titer Plates. Sold as case of 10 Cap-Mats or single 2231.

VIALS AND VIAL SETS

Vial sets are an alternative to titer plates. Vials can be filled and sealed individually, and the larger vials can hold bigger samples than titer plates. Replacement vials and steel balls can be purchased separately.

2240–PC 4 ML POLYCARBONATE VIAL SET

Each set includes 24 pre-cleaned 2241-PC vials, each with a 3/8 in. (9.5 mm) steel grinding ball 2155. Grinding load per vial 1.5 mL. Sold as case of 10.

2240–PEF 4 ML PRE-CLEANED FROSTED POLYETHYLENE VIAL SET

Set of 24 vials, each with one 3/8 in. (9.5 mm) steel grinding ball 2155. Grinding load per vial 1.5 mL. Sold as case of 10.

2241–PC 4 ML POLYCARBONATE VIAL

Polycarbonate vial with screw-on polyethylene cap. 1/2 in. diameter x 2 in. long (12.7 x 50.8 mm). Grinding load per vial 1.5 mL. Sold as case of 240.

2241–PEF-200 5 ML FROSTED POLYETHYLENE VIAL

1/2 in. diameter x 2 in. long (12.7 x 50.8 mm). Grinding load per vial 1.5 mL. Sold in units of 200.

2250 15 ML PRE-CLEANED POLYCARBONATE VIAL SET

Includes 5 pre-cleaned 2251PC vials pre-loaded with two 7/16 in. (11 mm) steel grinding balls 2156. Reinforced caps available 2250R. Sold as case of 10.

2251-PC 15 ML, SHORT VIAL POLYCARBONATE

Polycarbonate vial with screw-on cap, 1 1/8 x 1 2/3 in. (2.9 cm x 4.2 cm). Pre-cleaned with grinding load per vial 6 mL. Sold as bag of 100.

2252–PC-30 15 ML TALL VIAL POLYCARBONATE

Polycarbonate vial with screw-on cap, 5/8 in. x 4¾ in. (1.6 cm x 12.1 cm). Grinding load 6 mL. Sold as a pack of 30 vials.

2253-PC-48 50 ML POLYCARBONATE VIAL

Polycarbonate vial with screw-on cap, 1 1/8 in. x 4 ½ in. (2.9 cm x 11.4 cm). Grinding load 20 mL. Sold as a pack of 48 2253C-48.

2310 2 ML REINFORCED TUBE

Reinforced, Self-standing 2mL microfuge tube with screw-cap. Sold in packs of 200.

2254 75 ML JAR SET POLYCARBONATE

75 mL polycarbonate vial with with screw-on cap and rubber gasket. Supplied with two half inch steel balls. Sold in pairs.

2248 12 OZ. JAR
12 oz. (355 mL) PET jar. Sold in packs of 8.

2256 16 OZ. JAR
16 oz. (480 mL) PET jar with aluminum lined plastic cap. Sold in packs of 12.

2258 25 OZ. JAR
25 oz. (750 mL) PET jar. Sold in packs of 4.

PRE-LOADED 2 ML DISRUPTION TUBES

Ideal for fast and simple setup—add samples and you're ready to go. Vials are self-standing, making it easier to add samples, and are prefilled with your choice of molecular biology grade silica beads, acid washed silica or zirconia beads, silica beads or steel balls. Approx. grinding load per vial is 0.5–1.0 mL.

2301–100MB
2 mL microfuge tube with screw-cap, prefilled with 100 µm molecular biology grade silica beads. Sold in packs of 100.

2302–100AW2
2 mL microfuge tube with screw-cap, prefilled with 600 mg of 100 µm acid washed zirconia beads. Available as 200 µm 2302-200AW Sold in packs of 100.

2302–1700AW
2 mL microfuge tube with screw-cap, prefilled with 1.7 mm acid washed zirconia beads. Sold in packs of 50.

2302–3000AW
2 mL microfuge tube with screw-cap, prefilled with 3 mm acid washed zirconia beads. Sold in packs of 50.

2302–1000AW
2 mL microfuge tube with screw-cap, prefilled with 1.0 mm acid washed zirconia beads. Sold in packs of 100.

2302–1400AW
2 mL microfuge tube with screw-cap, prefilled with 1.4 mm acid washed zirconia beads. Sold in packs of 100.

2303–MM1
2 mL microfuge tube with screw-cap, prefilled with 800 µm and 1.4 mm acid washed zirconia beads. Sold in packs of 100.

2303–MM2
2 mL microfuge tube with screw-cap, prefilled with 800 µm and 1.4 mm acid washed zirconia beads. Sold in packs of 100.

2303–MM3
2 mL microfuge tube with screw-cap, prefilled with 100 µm silica beads, 1.4 mm zirconia beads and a 4 mm acid washed silica bead. Sold in packs of 100.

2304–100AW
2 mL microfuge tube with screw-cap, prefilled with 100 µm acid washed silica beads. Sold in packs of 100.

2304–400AW
2 mL microfuge tube with screw-cap, prefilled with 400 µm acid washed silica beads. Sold in packs of 100.

2304–800AW
2 mL microfuge tube with screw-cap, prefilled with 800 µm acid washed silica beads. Sold in packs of 100.

2305-2800SS
2 mL microfuge tube with screw-cap, prefilled with 2.8 mm stainless steel grinding balls. Sold in packs of 50.

2302-6000AW
2 mL microfuge tube with 6 mm Ceria Stabilized Zirconia beads. For grinding tough plant samples. Sold as a pack of 100.

SamplePrep

2300-500E
2 mL Polypropylene microfuge tube.
Sold as a pack of 500.

HOLDERS AVAILABLE FOR GENOLYTE

1210 GENOLYTE HOLDER FOR 2 ML VIALS

Holds six standard 2mL vials.

1211 GENOLYTE HOLDER FOR 5 ML VIALS

Holds four 2241-PEF polyethylene vials.

1212 GENOLYTE HOLDER FOR 7 ML VIALS

Holds two 2142 polyethylene vials.

1215 GENOLYTE HOLDER FOR METAL 5 ML VIALS

Holds one steel capped 5005 vial or one Tungsten Carbide capped 5006 vial.

1216 GENOLYTE HOLDER FOR 12 ML VIALS

Holds two 6133PC-T polycarbonate vials.

1217 GENOLYTE HOLDER FOR AGATE 3.5 ML VIALS

Holds one 3120 agate vial.

METAL VIALS FOR GENOLYTE

5005 STEEL CAPPED 5ML VIAL

Steel capped 5mL vial with one 5/16 in. (7.9 mm) steel ball.

5006 TUNGSTEN CARBIDE VIAL SET

Tungsten carbide capped vial set 5 mL, with one tungsten carbide ball

METAL BALLS FOR GENOLYTE

5005B STEEL GRINDING BALL 5/16 IN. (7.9 MM)

Steel grinding ball for 5005 vial.

5006B TUNGSTEN CARBIDE BALL 5/16 IN. (7.9 MM)

Tungsten carbide ball for 5006 vial.

PRE-LOADED DISRUPTION TUBES REFERENCE TABLE

PART NUMBER	2 ML VIAL SET	DETAILS
2301-100MB	100 µm Silica Beads	Economical bead for disrupting bacteria.
2302-1400AW	1.4 mm Zirconia Beads	Suitable for small tissue samples and biomass.
2303-MM1	Garnet & ZrO_2 Satellites	General Sample Shredding.
2303-MM2	800 micron & 1.4 mm Zirconia Beads	Mycelium & Soft Leaves.
2303-MM3	100 micron Si, 1.4 mm Zr, & 4 mm Si	Biofilms & Plant Tissues.
2304-100AW	100 µm Silica Beads (600mg)	Suitable for Bacteria.

2302-100AW2	100 µm Zirconia Beads	Zircomium beads are of higher density; excellent for bacterial disruption.
2304-400AW	400 µm Silica Beads	Size is ideal for yeasts such as Saccharomyces.
2304-800AW	800 µm Silica Beads	Size is suitable for molds and pollen.
2302-1000AW	1.0 mm Zirconia Beads	Beads are suitable to disrupt finer soils.
2302-1700AW	1.7 mm Zirconia Beads	Effective for larger tissue samples and fine plant materials.
2305-2800SS	2.8 mm Stainless Steel Grinding Balls	Most dense of all the grinding media, popular for their moderate cost. Good for tissues.
2302-3000AW	3.0 mm Zirconia Beads	Good for larger tissue samples. Excellent chemical resistance to organics.
2302-6000AW	6 mm Ceria Stabilized Zirconia Beads	Ideal for grinding tough plant samples.

RACKS AND HOLDERS

These Racks and Holders are designed to hold vials securely in place on the 2010 Geno/Grinder or MiniG during processing. They are lightweight and can also be used on the benchtop and to transport samples within the laboratory.

MiniG® ACCESSORIES

1680 2 ML FOAM HOLDER FOR MiniG®

Rugged foam block holds 24 standard 2 mL tubes. Sold as a pair.

1681 5 ML BLOCK RUGGED FOAM BLOCK

Holds 24 standard 5 mL tubes. Sold in pairs.

1685 15 ML FOAM HOLDER FOR MiniG®

Rugged foam block holds 12 standard 15 mL tubes.

1686 50 ML FOAM HOLDER FOR MiniG®

Rugged foam block holds six standard 50 mL tubes.

1688 FOAM HOLDER

Foam holder for 75 mL polycarbonate jars, holds 2 jars.

GENO/GRINDER ACCESSORIES

2191 5 ML VIAL HOLDER

Thick, rugged foam block holds 48 standard 5 mL centrifuge tubes.

2193 15 ML VIAL HOLDER

Holds up to 12 x 15 mL vials 2251-PC.

2196-16-PE HOLDER FOR 50 ML CENTRIFUGE TUBES

Thick, rugged foam block holds 16 standard 50 mL centrifuge tubes.

2197 HOLDER FOR 15 ML CENTRIFUGE TUBES

Thick, rugged foam block holds 24 standard 15 mL centrifuge tubes.

2300 HOLDER FOR 2 ML MICRO-CENTRIFUGE TUBES

Thick, rugged foam block holds 48 microfuge tubes.

2198 FOAM HOLDER FOR 75ML POLYCARBONATE JARS

Thick, foam block holds six 75 mL 2254 jars.

2257 FOAM HOLDER FOR 2255 VIALS

Thick, rugged polyethylene foam block holds up to sixteen 2255 vials.

2259 HOLDER FOR 2258 JARS

Foam holder for two 25 oz. (750 mL) jars.

KRYO-TECH® ACCESSORIES

These accessories are used to chill samples to cryogenic temperature and to maintain temperature during grinding in the Geno/Grinder®. Additional products for liquid nitrogen handling, such as protective gloves and portable Dewars, can be found in the Freezer/Mill® section.

2600 CRYO-STATION

The Cryo-Station holds temperature sensitive samples that must be kept chilled for cryogenic grinding or to preserve pesticides, RNA, or proteins for extraction. It has an insulated jacket and can be filled manually or automatically with liquid nitrogen. The well of the Cryo-Station reaches liquid nitrogen temperatures quickly. Up to two Cryo-Blocks can be placed in the well to be chilled and kept cold prior to grinding. The well can also hold a chilled cutting board to prepare temperature sensitive samples.

2255 CRYOGENIC GRINDING VIAL PACK

Used to grind tough materials (e.g. bone) at cryogenic temperatures. Package includes four sets of cryogenic vials, each with 1 cylinder, 2 end plugs, and 2 tungsten carbide balls. For use with the 2257 holder or the 2260 Cryo-Block. Sold in a pack of 4.

2650 CRYO-ADAPTER FOR TITER PLATES

Extruded aluminum insert for 2210 Titer Plate. Keeps samples in titer plate cold during grinding. Sold in pairs.

2189C CRYOPLATE

THE 2189C CRYOPLATE keeps sample lids cold during the grinding process. Used with Cryo-Block or 2650 titer plates.

CRYO-BLOCKS

Aluminum Cryo-Blocks are designed to keep sample tubes or vials cold during processing. They can be pre-cooled in liquid nitrigen, dry ice or a freezer. Cryo-Blocks should always be used two at a time to balance the load in Geno/Grinder Clamps.

Speed restrictions may apply for heavier loads, please see the technical info on page 36.

MiniG® CRYO-BLOCKS

1660 2 ML CRYO-BLOCK

Lightweight cryo-block for 2 ml vials with a hollow base and a lid to help the samples maintain cold temperature during processing.

1665 5 ML CRYO-BLOCK

Lightweight cryo-block for 5 ml vials with a hollow base and a lid to help the samples maintain cold temperature during processing.

1666 CRYO-BLOCK FOR 15 ML CENTRIFUGE TUBES

Aluminum block that holds fifteen 15 mL round-bottom centrifuge tubes.

1667 CRYO-BLOCK FOR 50 ML CENTRIFUGE TUBES

Aluminum block that holds six 50 mL round-bottom centrifuge tubes.

1668 CRYO-BLOCK FOR 15 ML POLYCARBONATE VIALS

Aluminum block that holds six 2251PC vials.

GENO/GRINDER® CRYO-BLOCKS

2661 CRYO-BLOCK FOR 15 ML CENTRIFUGE TUBES

Aluminum block that holds fifteen 15 mL round-bottom centrifuge tubes. Sold in pairs.

2660 CRYO-BLOCK FOR 15 ML POLYCARBONATE VIALS

Aluminum block that holds six 2251PC vials. Sold in pairs.

2663 CRYO-BLOCK FOR 5 ML POLYETHYLENE VIALS

Aluminum block that holds twenty-four 2241-PEF vials, as used in 2240-PEF Vial Set. Sold in pairs.

2662 CRYO-BLOCK FOR 5 ML POLYCARBONATE VIALS

Aluminum block that holds twenty-four 2241-PC vials, as used in 2240-PC Vial Set. Sold in pairs.

2665 CRYO-BLOCK FOR MICRO-CENTRIFUGE OR PCR TUBES

Aluminum block that holds 96 standard (0.6 mL) micro-centrifuge tubes. Sold in pairs.

2664 CRYO-BLOCK FOR 50 ML CENTRIFUGE TUBES

Aluminum block that holds six standard conical-bottom 50 mL centrifuge tubes for cryogenic milling. Sold in pairs.

2666 CRYO-BLOCK FOR 48 MICRO-CENTRIFUGE OR PCR TUBES

Aluminum block that holds 48 standard (1.5 - 2.0 mL) micro-centrifuge tubes. Sold in pairs.

2260 CRYO-BLOCK FOR 2255 VIAL

Aluminum block holds eight 2255 vials. Two Cryo-Blocks, allows 16 samples to run simultaneously. Sold in pairs.

TECHNICAL INFORMATION

VIAL AND TITER PLATE CAPACITY TABLE

	PART #	MiniG CAPACITY	HOLDER	NUMBER OF LAYERS
Titer plate	Various	2	n/a	2
2 mL vial	Various	48	1680	2
5 mL vial	Various	48	1681	2
15 mL vial (short)	Various	10	n/a	2
15 mL vial (tall)	2252-PC-30	12	1685	1
50 mL vial (tall)	2253-PC-48	6	1686	1
75 mL vial	2254	2	1688	1
Cryovials	2255	n/a	n/a	1
16 oz. (480 mL) jar	2256	1	n/a	1
25 oz. (750 mL) jar	2258	n/a	n/a	1

STACKING TRAY	GENO/ GRINDER CAPACITY	HOLDER	NUMBER OF LAYERS	STACKING
1690T	6	n/a	3	2189T
1690T	96	2300	2	2189C
1690T	96	2191	2	2189C
1690T	24	2193	2	2189C
n/a	24	2197	1	n/a
n/a	16	2196-16-PE	1	n/a
n/a	6	2198	1	n/a
n/a	6	2257	1	n/a
n/a	2	n/a	1	n/a
n/a	2	2259	1	n/a

OPERATING LOAD RESTRICTIONS

To maintain proper functionality of the Geno/Grinder, the maximum recommended total sample load in the clamp assembly is 4 lb. (1.8 kg). The total sample load includes sample, vials (or titer plates), grinding media, holders (or cryo-blocks), and nesting trays. For Sample loads exceeding 2.0 lb.

(0.9 kg), the maximum recommended operating rate is 1500 rpm. Sample loads less than 2.0 lb. can be run at rates up to the maximum of 1750 rpm. Dynamic loads greater than 2 lb. can create a rate error. When stacking 4 or 6 titer plates, do not add more than five 4 mm steel balls to a single titer plate well.

Operating with loads that exceed the recommended maximum rate and weight limits can result in damage to the Geno/Grinder. Therefore, warranty restrictions or invalidation may apply.

USING THE GENO/GRINDER® FOR BEAD BEATING

Bead beating is the preferred method to disrupt a variety of microorganisms and plant or animal tissues. In bead milling, grinding media such as steel or ceramic balls or glass beads are vigorously agitated inside a sealed vial or titer plate with the sample. The most commonly used grinding balls are steel ball bearings 4 mm in diameter. Disruption or cell lysis occurs as a result of the crushing action of the grinding media as they collide with the sample. Low shearing of nucleic acids can be achieved by varying the agitation speed of the mill. It is considered the method of choice for disruption of yeast and fungi and tough-to-disrupt cells such as bacteria and microalgae.

This method is one of the few that avoids cross-contamination between samples because both vials and grinding media are disposable. SPEX SamplePrep offers a comprehensive range of grinding media, vials, jars and titer plates for ranging from 0.6 to 750 mL. The size and amount of the grinding media used is important. Speed and effectiveness of disruption can be increased dramatically by increasing the density, form and amount of grinding media in the sample vial or titer plate. Tough tissues may require precooling to embrittle the sample and this also serves to preserve any temperature-sensitive components such as RNA or Proteins. SPEX SamplePrep offers a full range of cryogenic Kryo-Tech® accessories for this purpose. The loading of the beads should always allow adequate movement inside the sample vials but can be up to 80% of the total sample volume, provided there is still adequate agitation

of the bead slurry. The higher the ratio of grinding media to sample volume, the faster the rate of cell disruption. After homogenization, the beads settle and the cell extract can be removed.

The Geno/Grinder® offers analysts a versatile high-throughput bead mill for plant or animal tissue homogenization. The patented design has a true linear grinding motion that provides the most efficient mechanism for tissue homogenization and cell lysis. The powerful and compact design enables complete disruption with or without buffer in about 1-3 minutes, with high yields. The Geno/Grinder® has also been successfully used for pesticide residue extraction from fruit and vegetable samples using the QuEChERS technique.

A comprehensive guide for beating is available. For further information visit www.spexsampleprep/application-notes.

PROTOCOLS


BEFORE


AFTER

SPELT SEEDS BEAD BEATING

Insert one seed and one grinding ball in each well of the deep well titer plate.

Grinding Balls=4 mm
Rate=1,400 RPM
Time=1 minute


BEFORE


AFTER

CANNABIS

Place two grinding balls into a 50 mL centrifuge tube.

Grinding Balls=11 mm
Rate= 1,500 RPM
Time= 1-2 minutes


BEFORE


AFTER

RICE

Dispense one steel bead into each well of the deep well titer plate.

Grinding Balls=4 mm
Rate= 1,500 RPM
Time= 2 minutes

BEFORE & AFTER SAMPLES

CORN

Sample was inserted in 2255 vials with two tungsten carbide balls (7/16"). 15 minute pre-cool of vials in Cryo-Block in liquid nitrogen. Five minute grind at 1,500 RPM.

BEFORE

AFTER

FRUIT

Insert pre-homogenized fruit into 50 mL tubes. Insert into foam holders, place into clamp and run Geno/Grinder from 1,000 to 1,500 RPM for 1 minute with solvent or water.

BEFORE

AFTER

ANIMAL TISSUE

Pre-chill sample in 2251 grinding container using a 2660 Cryo-block submerged in liquid nitrogen for 5 minutes. Set Geno/Grinder speed for 1,500 RPM for 2 minutes.

BEFORE

AFTER

APPLICATION NOTES

APPLICATION NOTE SP001: CELL DISRUPTION / APPLICATION: CELL LYSIS

MECHANICAL DISRUPTION FOR HIGH-THROUGHPUT FATTY ACID EXTRACTION FROM ANIMAL TISSUE SAMPLES

Sample preparation is often the bottleneck in the process of efficient analysis work. Up to now efforts to mechanically disrupt cells through grinding have been based on the modification of traditional ball or swing mills with an adapter for micro titer plates. The Geno/Grinder® was the first available mill specifically designed for cell disruption. Two deep well plates (PP), used for collecting and storing of biological materials with 1.5 – 2 ml wells were placed next to each other in the Geno/Grinder clamp assembly. Freeze dried rat muscle or livers were placed in each plate well and spiked with 100 mm KH_2PO_4 buffer at pH 2 or trichloro-acetic acid.

APPLICATION NOTE SP011: HOMOGENIZATION APPLICATION: DNA EXTRACTION OF LEAF MATERIAL

EXTRACTION OF NUCLEIC ACIDS FROM SUGAR BEET LEAVES

Sugar beet leaf tissue is freeze-dried, and 0.5 to 1 grams of each sample is placed in a 2mL Eppendorf vial with two steel balls. The samples are pulverized, 96 at a time, in a SPEX Geno/Grinder that is run for two periods of one minute at 1000 strokes per minute. Then 300 µL of buffer is added to each vial and the contents homogenized by running the vials for 30 seconds at 1000 strokes/minute. The vials are centrifuged and the lysed plant tissue transferred to microtiter plates, then mixed, filtered, rinsed and dried prior to DNA analysis.

APPLICATION NOTE SP013: GRINDING / DISRUPTION APPLICATION: STARCH AND SOLUBLE METABOLITE MEASUREMENT IN LEAVES

PERCHLORIC ACID EXTRACTION OF LEAVES FOR MEASUREMENT OF STARCH AND SOLUBLE METABOLITES

Harvested plant material is placed in SPEX 5-mL vials and chilled in liquid nitrogen. Perchloric acid is added to each vial along with a stainless steel ball. The vials are run in a SPEX Geno/Grinder for 90 seconds at 1500 strokes per minute, or longer if necessary to pulverize the contents. More perchloric acid is added to each vial, which is mixed, then centrifuged. The supernatant is analyzed for soluble metabolites, and the centrifuged pellet assayed for starch.

APPLICATION NOTE SP014: RNA EXTRACTION FROM FUNGUS APPLICATION: RNA EXTRACTION

RNA EXTRACTION FROM ASPERGILLUS PARASITICUS MYCELIUM

Some strains of the filamentous fungus Aspergillis Paraciticus are nontoxic, and others produce aflatoxins in infected grain and other food products. To study this, RNA was extracted from Aspergillis samples after pulverizing them cryogenically in a SPEX Geno/Grinder, then adding Puresol to the vials and returning them to the Geno/Grinder to homogenize the samples.

APPLICATION NOTE SP016: LYSING / HOMOGENIZATION APPLICATION: DNA EXTRACTION FROM RICE SEEDS

QUICK DNA EXTRACTION FROM RICE SEED

Samples are prepared using a 96-well 1 mL assay block. Dispense one 5/32 in. (4 mm) stainless steel bead into each well using the Grinding Ball Dispenser (SPEX SamplePrep Cat. No. 2100). Next, add one seed to each well. Dispense extraction buffer into each well and securely cap each well. After the samples have been capped, grind them in the Geno/Grinder at 500 strokes/minute for two minutes.

APPLICATION NOTE SP017: LYSIS TIME AND OTHER VARIABLES ON DNA EXTRACTION / APPLICATION: DNA EXTRACTION FROM FRESH BASIL LYSED

EFFECT OF LYSIS TIME AND OTHER VARIABLES ON DNA EXTRACTION FROM FRESH BASIL LYSED IN 2 ML TUBES WITH THE GENO/GRINDER®

The Geno/Grinder® was compared with a competitive cell lyser (Competitor A) for extraction of DNA from fresh basil. In addition, homogenization time and operating rate were varied for the Geno/Grinder, while buffers, grinding media and tube size remained constant. Results indicated that use of the Geno/Grinder provided DNA with higher molecular weight than the competitive instrument. In addition, optimal homogenization conditions using the Geno/Grinder were found to be 90 sec. at a rate of 2000 cyc/min.

APPLICATION NOTE SP018: HIGH THROUGHPUT DISRUPTION OF YEAST IN A 96-WELL FORMAT / APPLICATION: DNA/RNA AND OTHER EXTRACTIONS

HIGH THROUGHPUT DISRUPTION OF YEAST IN A 96-WELL FORMAT

Mechanical disruption of yeasts has traditionally been accomplished by using either a french press or bead beater. In both approaches, samples are processed individually. For experiments where large numbers of yeast clones must be examined in a high throughput screening environment, individual processing is a major bottleneck and impractical. Consequently, a method is needed that combines mechanical disruption of cells in a high throughput format. The Geno/Grinder (SPEX SamplePrep, Metuchen, NJ), a bead beater originally designed to smash seeds in deep well plates, can be used to disrupt yeast in a microwell plate format.

APPLICATION NOTE SP020: LYSING OF BACTERIAL CELLS APPLICATION: DNA/RNA AND OTHER EXTRACTIONS

LYSING OF BACTERIAL CELLS IN THE GENO/GRINDER®

The Geno/Grinder was tested to determine if this technology could be used to lyse bacterial cells. Standard 96-well titer plates were used with 400-600 µ silica grinding beads (Molecular Biology Grade, cat. no. 2166). The delivery of the beads into each cell of the titer plate can be accomplished in a number of ways. In this case micropipette tips were filled to the mark with grinding beads, and each tip emptied into a titer plate well. This technique will deliver approximately 0.4 grams of silica beads per well. Other delivery systems are commercially available.

APPLICATION NOTE SP021: EXTRACTION OF RNA/CDNA AND GENOMIC DNA FROM TISSUE WITH REAL-TIME PCR / APPLICATION: DNA/RNA AND OTHER EXTRACTIONS

EXTRACTION OF RNA/CDNA AND GENOMIC DNA FROM TISSUE WITH REAL-TIME PCR

Fresh samples of animal tissue were collected, trimmed to approximately 50–100 mg of wet weight, snap-frozen in liquid nitrogen, and stored at –80° C. The animals were man, dog, cat, mouse, cow, and horse, as well as fish and clams; see Table 1. Before DNA and/or RNA extraction, the tissues were transferred frozen to a deep-well titer plate standing on a block of dry ice. Each well contained two 4 mm stainless steel balls (SPEX CertiPrep cat. no. 2150) and 500 microliters of buffer (Applied Biosystems nucleic acid purification lysis buffer). The plates were sealed with a plastic cover and subjected to grinding in the 2000 Geno/Grinder for 2 minutes at a setting of 1000 strokes/minute.

APPLICATION NOTE SP027: SAMPLE HOMOGENIZATION OF FOOD SAMPLES FOR STABLE ISOTOPE RATIO ANALYSIS

STABLE ISOTOPE RATIO ANALYSIS FOR THE TESTING OF AUTHENTICITY OF FOOD PRODUCT ORIGIN AND PRODUCTION METHOD CLAIMS APPLICATION

Food Fraud is a global problem with a long heritage. With global food supply chains growing in complexity, fraudulent food labelling has become an ever increasing risk. Consumers are becoming more aware and concerned with how and where their food products originate. Suppliers are taking a more active interest in protecting their brand reputation. Through the application of Stable Isotope Ratio Analysis (SIRA), consistency with labelling claims for geographical origin and/or production method of food and food products can be established. By using this technique we are able to help protect consumer, supplier and producer interests.

APPLICATION NOTE SP024: PESTICIDE ANAYLSIS: STANDARD QUECHERS VS MODIFIED METHOD / APPLICATION: QUECHERS / PESTICIDE EXTRACTION

ANALYSIS OF PESTICIDES IN FRUIT AND VEGETABLE PRODUCTS USING A STANDARD QUECHERS METHOD AND A MODIFIED METHOD INVOLVING THE GENO/GRINDER

Pesticide residues in agricultural food sources are widely considered to cause adverse health effects when consumed by humans. In particular, much of the produce sold in the U.S. is imported and concern over pesticide levels in these fruits and vegetables in comparison to those grown domestically has resulted in increased testing for pesticide residues.

In this study, the Geno/Grinder was employed to homogenize the fruit/vegetable samples and to mix the produce rapidly and thoroughly with the salts and solvent in an effort to improve the extraction step. The goal of the study was to determine whether the use of the Geno/Grinder during the extraction step would increase pesticide recovery over the traditional, manual QuEChERS method.

APPLICATION NOTE SP022: THE BENEFITS OF THE GENO/GRINDER FOR PESTICIDE RESIDUE ANALYSIS / APPLICATION: PESTICIDE RESIDUE EXTRACTION

THE BENEFITS OF THE GENO/GRINDER®HIGH-THROUGHPUT TISSUE HOMOGENIZER TO INCREASE SAMPLE THROUGHPUT FOR PESTICIDE RESIDUE ANALYSIS BY LC/MS/MS

Sample preparation for pesticide residue analysis has typically followed DuPont Report No. AMR 3705-95, "Analytical Method for the Determination of Famoxadone and Cymoxanil Residues in Various Matrices." (2) In this procedure, ground-up samples are weighed into extraction jars, followed by the addition of water to allow them to re-hydrate prior to extraction. Acetonitrile is added and samples are ground with a laboratory homogenizer. The plant matrix is then allowed to settleout and liquid extracts are filtered and collected in mixing cylinders containing sodium chloride. The mixing cylinders are capped, shaken, and inverted to aid in the dissolution of sodium chloride and then allowed to stand while the acetonitrile (upper layer) and water phases separate. Acetonitrile aliquots are taken for cymoxanil and famoxadone analysis.

Canna-Prep®

Cannabis Sample Preparation

SPEX SamplePrep is an industry leader in Sample Preparation products for the Cannabis Industry. Our Homogenizers, Grinders and Cryogenic Mills provide reliable, reproducible results with no cross contamination. We have the expertise and equipment to help startups and established labs.

To learn more about how our products can be used in cannabis testing and applications visit our website www.canna-prep.com.

GENO/GRINDER®

FREEZER/MILL®

Scan the QR code with your smart phone to see how our Freezer/Mill easily grinds edibles.

CRYOGENIC MILLS

Our programmable cryogenic mills are specifically designed for grinding tough and/or temperature sensitive samples. Sample vials are continuously cooled with liquid nitrogen while samples are pulverized by magnetically shuttling a steel impactor back and forth against the vial end-plugs. Since the vials are closed, the integrity of the contents is maintained, hazardous or critical samples are easily controlled, cleanup is simplified and cross contamination is minimized. An intuitive touch screen control panel allows users to save up to 20 grinding protocols.

The **6775 Freezer/Mill®** is a compact cryogenic grinder that accommodates samples from 0.1 to 5 g.

The **6875 Freezer/Mill®** is a high capacity cryogenic grinder that accommodates samples up to 100 g.

The **6875D Freezer/Mill®** is a dual chamber high capacity cryogenic grinder that accommodates samples up to 100 g per chamber.

The Freezer/Mill can grind almost every natural biological plant or animal based material to a fine particle size to enable fast and efficient extraction of nucleic acids, proteins and other molecules. Freezer/Mills can also grind samples that are virtually impossible to grind at room temperature such as polymers.

APPLICATIONS

DNA/RNA extractions
Yeast analysis
Pharmaceutical analysis
Drug testing
RoHS/WEEE
Food Testing
Consumer product testing
Materials research

SAMPLE TYPES

Plant/Animal tissue
Cannabis/Edibles
Polymers
Yeast
Pharmaceuticals
Food products
Electronic components
Textiles
Hair
Bone

6775 FREEZER/MILL®

The 6775 Freezer/Mill® is a compact cryogenic mill that accommodates samples ranging from 0.1-5g. Its grinding chamber holds one small grinding vial or one microvial set. It's specifically designed for the cryogenic grinding of tough and/or temperature sensitive samples in a grinding vial immersed in liquid nitrogen throughout the grinding cycle.

SPECIFICATIONS

VOLTAGE	100–120 V, 60 / 50 Hz 200–240 V, 50 / 60 Hz
DIMENSIONS	19 in. (48 cm) x 10.5 in. (27 cm) x 12.75 in. (32 cm)
WEIGHT (LBS)	22 lbs. (10 kg)
TIMER/CONTROLS	Stores up to 20 grinding programs
POWER CORD	Supplied with a 3-prong grounded cord 115 V, 60 Hz, or a 2-prong grounded European cord for 230 V, 50 Hz.

GRINDING OPTIONS

- Small vials
- Microvials

- Tiltable control panel

- Easy to load

6875 & 6875A FREEZER/MILL®

The 6875 Freezer/Mill® is a high capacity cryogenic mill that accommodates up to 100 grams of sample. It's specifically designed for cryogenic grinding of pulverizing tough and/or temperature sensitive samples in a grinding vial immersed in liquid nitrogen.

Touch-screen controls for programming grinding cycles and time, impact rate, pre-cooling and cooling times.

Lid safety interlock for operator protection.

Magnetically driven impactor in the vial is the only moving part.

Chamber holds one large vial, one mid-sized vial, or up to four small vials or four microvial sets.

Insulated liquid nitrogen tub is equipped with LN level sensor.

Auto-Fill system available (6875A)

SPECIFICATIONS

VOLTAGE	100–120 V, 60 / 50 Hz 200–240 V, 50 / 60 Hz
DIMENSIONS	20.5 in (52 cm) x 21.5 in (55 cm) x 18 in (46 cm)
WEIGHT (LBS)	44 lbs (20 kg)
TIMER/CONTROLS	Stores up to 20 grinding programs
POWER CORD	Supplied with a 3-prong grounded cord 115 V, 60 Hz, or a 2-prong grounded European cord for 230 V, 50 Hz.

GRINDING OPTIONS

- Easy to load

- Up to four small vials or 12 microvials

- One mid-size vial
- One large vial

6875D FREEZER/MILL®

The 6875D Freezer/Mill® is a high capacity cryogenic mill that has dual grinding and cooling chambers. It allows you to pre-cool one set of samples while another set is being ground. Dual grinding chambers can grind up to 200 grams in a single run. (100 grams per chamber.)

Touch-screen controls for programming grinding cycles and time, impact rate, pre-cooling and cooling times.

Lid safety interlock for operator protection.

Dual pre-cooling chambers maintains samples at cryogenic temperature while other samples are grinding.

Magnetically driven impactor in the vial is the only moving part.

Each grinding chamber holds one large grinding vial or one mid-sized vial or up to four small grinding vials or four microvial sets.

Insulated liquid nitrogen tub is equipped with LN level sensor.

Equipped with Auto-Fill system

SPECIFICATIONS

VOLTAGE	100–120 V, 60 / 50 Hz 200–240 V, 50 / 60 Hz
DIMENSIONS	20 in (50.8 cm) x 21 in (53.34 cm) x 25.5 in (64.77 cm)
WEIGHT (LBS)	75 lbs. (34 kg)
TIMER/CONTROLS	Stores up to 20 grinding programs
POWER CORD	Supplied with a 3-prong grounded cord 115 V, 60 Hz, or a 2-prong grounded European cord for 230 V, 50 Hz.

MIXING & GRINDING OPTIONS

- One mid-size or large vial per grinding chamber

- Up to four small vials or 12 microvials per chamber

- Dual pre-cooling chambers

VIAL SET REFERENCE

6775 FREEZER/MILL® - ONE CHAMBER - SAMPLE WEIGHT: 0.1 - 5 g

VIAL SETS

6751 - Small Grinding Vial Set	6771 - Small Low-chrome Grinding Vial Set	6757 - Microvial Set
6761 - Small Poly-Vial Set	6781S - Stainless Steel Grinding Vial Set	

VIAL EXTRACTORS	ACCESSORIES	
6756 - Extractor For Small Vials	6905 - Portable Cryogenic Dewar - 25L	6906 - Short Cryogenic Transfer Hose, 4 Ft (1.2 M)
6758 - Extractor For Microvials	6907 - Long Cryogenic Transfer Hose, 6 ft (1.8m)	

6875 & 6875D FREEZER/MILL® - ONE OR TWO CHAMBER - SAMPLE WEIGHT: - UP TO 100 G PER CHAMBER

VIAL SETS

6751 - Small Grinding Vial Set	6771 - Small Low-chrome Grinding Vial Set	6881 - Mid-Size Grinding Vial Set
6757 - Microvial Set	6781S - Stainless Steel	6883 - Mid-Size Low-chrome Grinding Vial Set
6761 - Small Poly-Vial Set	6801 - Large Grinding Vial Set	6885 - Mid-Size Poly-Vial Set

VIAL HOLDERS

6807 - Multi-Vial Holder for small vials	6888 - Multi-Vial Holder for poly carb mid-size vials	6887 - Multi-Vial Holder for mid-size vials

VIAL EXTRACTORS	ACCESSORIES	
6756 - Extractor For Small Vials	6808M - Mid-Size Vial Adapter Kit	6907 - Long Cryogenic Transfer Hose, 6 ft (1.8m)
6758 - Extractor For Microvials	6870M - Medium Grinding Vial Accessory Package	6906 - Short Cryogenic Transfer Hose, 4 Ft (1.2 M)
6808 - Extractor For large/ mid- size Vials	6870L - Large Grinding Vial Accessory Package	6870S - Grinding Vial Accessory Package

FREEZER/MILL® GRINDING VIAL GUIDE

FREEZER/MILL®	MICROVIAL, SET OF 3 (0.1–0.5 G EACH)	SMALL VIAL (0.1–5 G)	MEDIUM VIAL (1–40 G)	LARGE VIAL (1–100 G)
6775 Freezer/Mill®	1 Set (3 total)	1	N/A	N/A
6875 Freezer/Mill®	4 Sets (12 total)	4	1	1
6875D Freezer/Mill®	8 Sets (24 total)	8	2	2

FREEZER/MILL® ACCESSORIES

GRINDING VIALS

Vials for the Freezer/Mill product line consist of a center cylinder, two end plugs, and an impactor. The center cylinder forms the grinding chamber, while the end plugs are removable for extraction of sample, and cleaning. The solid, cylindrical impactor is magnetically shuttled from end to end in the vial, with grinding occurring as the sample is struck by the impactor against the end plugs. Sample capacity is approximate. Actual sample capacity range for a vial depends on the nature and density of the material. Polycarbonate center cylinders allow visual checks of grinding progress.

6751 SMALL GRINDING VIAL SET

Set includes stainless steel impactor, 2 end plugs, and 4 polycarbonate center cylinders. Sample capacity 0.5 - 4.0 mL (0.1 - 5.0 g).

6751C4 SMALL POLYCARBONATE CENTER CYLINDER

For 6751 vial set; sold in package of 4. Also available as a pack of 20 6751C20.

6757 MICROVIAL SET

3 microvials in a vial holder allowing simultaneous sample processing. Each vial holds up to 0.5 g of samples. Ideal for grinding small quantities of samples such as plant and animal tissue.

6761 SMALL POLY-VIAL SET

Polycarbonate end plugs and cylinder, polycarbonate encapsulated steel impactor. Includes one complete vial and 3 spare center cylinders. Sample capacity 0.5 - 4.0 mL (0.1–5.0 g).

6771 SMALL LOW-CHROME VIAL SET

End plugs and impactor are low-chrome ASTM 06 steel. Set includes 6751C cylinders. Sample capacity 0.5–4.0 mL (0.1–5 g).

6781S SMALL STAINLESS STEEL GRINDING VIAL SET

Set includes stainless steel impactor, stainless steel center cylinder, two end plugs, and pack of ten O-rings.

6801 LARGE GRINDING VIAL SET

Set includes stainless steel impactor and two end plugs, plus four polycarbonate center cylinders. Sample capacity up to 50 mL (1 - 100 g)

6801C4 LARGE POLYCARBONATE CENTER CYLINDER

Replacement polycarbonate center cylinders for 6801 Large Grinding Vial Set. Sold as a pack of 4 and 20 6801C20.

6803 LARGE STEEL VIAL SET

This grinding Vial set includes a stainless steel center cylinder, impactor and 2 end plugs and a packet of O-rings. Sample capacity of 50 mL (1-100g)

6871 LARGE LOW-CHROME VIAL SET

For grinding electronic components to test for chromium under RoHS/WEEE directives. Includes 1 complete vial plus 3 extra cylinders. Sample capacity up to 50 mL (1–100 g).

6881 MID-SIZE VIAL SET

Stainless steel end plugs and impactor, polycarbonate center cylinder 6881C. Sample capacity up to 25 mL (1–40 g). Set includes one complete vial plus three extra cylinders.

6883 MID-SIZE LOW-CHROME VIAL

One 6883P mid-size steel impactor, two 6883E mid-size steel end plugs and one pack of 6811C4 mid-size polycarbonate center cylinders. Sample capacity up to 25 mL (40 g)

6881C4 MID-SIZE POLYCARBONATE CENTER CYLINDER

Replacement polycarbonate center cylinders for 6881, 6883 and 6885 vial sets. Sample capacity up to 25 mL (1–40 g). Sold in packs of 4 and 20 6881C20.

6885 MID-SIZED POLY-VIAL

End plugs and center cylinder are polycarbonate, impactor is polycarbonate-encapsulated steel. Sample capacity up to 25 mL (1–40 g).

6881, 6883 & 6885 vials require adapters

CLEANING THE FREEZER/MILL VIALS

The polycarbonate cylinders (PC) are very durable and can withstand repeated use. They should always be inspected before use, and discarded if any cracks are visible. Cleaning them immediately after each use with detergent and hot water is a good laboratory practice. To sterilize PC center cylinders, rinse in a 10% bleach solution.

Some known chemicals not compatible with polycarbonate include acetone, alcohols, organic solvents (chloroform, ammonia), Diethylpyrocarbonate (DEPC), and phenols. Polycarbonate cylinders should not be autoclaved as this will weaken the cylinders (heating above 80 °C damages PC). Avoid washing with caustic solution as damage or cracking can occcur.

Stainless steel parts should be washed with detergent and hot water, then immediately dried. To sterilize stainless steel, wipe down with alcohol.

VIAL RACKS

6755 VIAL RACK FOR SMALL VIALS

Glass-reinforced acetal rack holds up to sixteen vials for storage and handling.

6805 VIAL RACK FOR LARGE VIALS

Epoxy-coated steel rack holds six 6801 or 6871 vial sets for storage and handling.

EXTRACTORS

Vial extractors allow users to remove cold end plugs from extractors in seconds. Simply place the vial into the extractor cradle and press down the extractor handle to remove the end plug of the vial. The sample can be retrieved from the vial while it's still cold. Extractors for microvials, small vials and large vials are available. Each extractor has suction cups that secure the extractor to the bench top allowing for one-handed operation. Visit our website to see videos of our extractors in action.

6756 EXTRACTOR FOR SMALL VIALS

This end plug extractor removes end plugs from small grinding vials in seconds. It can be used to open 6751, 6761, 6771 and 6781S vials.

6758 EXTRACTOR FOR MICROVIALS

This end plug extractor removes end plugs from chilled 6757 microvial sets in seconds.

6808 VIAL EXTRACTOR

The 6808 vial extractor opens large grinding vial sets 6801, 6871 used in large Freezer/Mills. Mid-Size vials 6881, 6883, 6885 can also be opened using the optional 6808M Mid-size Vial Adapter Kit.

6808M MID-SIZED VIAL ADAPTER KIT

This adapter works with the 6808 large vial extractor. When attached it allows the extractor to remove the end plugs from mid-size vials.

6754 EXTRACTOR/ VIAL OPENER FOR SMALL VIALS

Lifts small grinding vials in and out of all Freezer/Mills and removes end plugs.

6804 EXTRACTOR/ VIAL OPENER FOR LARGE AND MID-SIZED VIALS

Lifts large grinding vials in and out of all Freezer/Mills and removes end plugs with the help of lever.

6753C MICROVIAL SAMPLE EXTRACTION TOOL

Sample extraction tool for removing ground samples from 6757 microvials.

6884 MID-SIZE VIAL ADAPTER

Insert for bell of 6804 extractor to allow end plug extraction for mid-size vials. Easily installed and removed.

VIAL ADAPTERS & HOLDERS

6807 MULTI-VIAL HOLDER

Vial holder positions one to four small vials 6751, 6761, 6771, 6781S. It also holds the 6757 microvial set for 6875, and 6875D Freezer/Mills.

6888 MID-SIZED VIAL ADAPTER

Vial holder for standard mid-sized vials with polycarbonate end plugs used in the 6875 and 6875D Freezer/Mill.

6887 MID-SIZED VIAL ADAPTER

Vial holder for standard mid-sized vials with stainless steel end plugs 6881 and 6883.

ACCESSORY PACKAGES

Each 6875 and 6875D Freezer/Mill is supplied with your choice of either a 6870L large vial accessory package or a 6870S small vial accessory package. The 6870M mid-size accessory package is also available as an option; descriptions of all three accessory packages follow. Note that the 6875 and 6875D Freezer/Mills can all hold small, mid-size, and large vials, and if you are going to use more than one size of vial you will need the accessory packages for each size vial. All the accessory packages are available for separate purchase, as are the individual components of each accessory package.

6870S SMALL VIAL ACCESSORY PACKAGE

Includes 6807 Multi-Vial Holder, 6754 Extractor/Vial Opener, and 6755 Vial Rack.

6870M-1 MID-SIZE VIAL ACCESSORY PACKAGE

Includes 6884 Mid-Size Vial Adapter for 6804 Extractor/ Vial Opener, and 6887 Mid-Size Vial Adapter. Used in 6875 and 6875D Freezer/Mill.

6870L LARGE VIAL ACCESSORY PACKAGE

Includes 6804 Extractor/Vial Opener and 6805 Vial Rack.

LIQUID NITROGEN HANDLING

Our products are designed to aid the user in the safe handling of liquid nitrogen. Liquid nitrogen (LN) can be hazardous. Its boiling point is -195.8 °C (-320.4 °F) and it should be used in a well ventilated area.

When working with liquid nitrogen directly or indirectly, the LN Tank valve or hose, or chilled Freezer/Mill components, cryogenic gloves must be worn to protect hands. A face Shield is also recommended to protect eyes from possible splashing. Be careful not to splash liquid nitrogen onto clothes or unprotected skin.Wear proper protection equipment (PPE) and consult your local LN supplier for additional safety guidelines.

AUTO-FILL 6875A & 6875D FREEZER/MILL®

The Auto-Fill Freezer/Mills are designed to automatically maintain the required level of liquid nitrogen during operation. The Auto-Fill System is a more efficient and safer way to handle the volume of liquid nitrogen required for processing samples in the large Freezer/Mill. This automated system reduces the loss of liquid nitrogen and saves the time lost when manually filling the reservoir.

The Auto-Fill System requires a cryogenic transfer hose, such as the SPEX SamplePrep 6906 or 6907 Cryogenic Transfer Hoses, which are sold separately. Transfer hoses are available in different lengths and can be constructed with different materials. The transfer hose must be configured with a 1/2 inch (12.7 mm) JIC female flare nut at each end for proper installation. The 6875A includes an adapter to allow compatibility with the 6906 and 6907 Cryogenic Transfer Hoses.

The Auto-Fill System is also supplied with a safety relief valve that connects to the liquid nitrogen supply via a 1/2 inch (12.7 mm) JIC female flare nut. The safety relief valve attaches at the high point of the transfer hose, and vents nitrogen gas pressure that may build up in the hose when there is liquid nitrogen in the hose and the valves at both ends are closed.

6900 CRYOGENIC GLOVES

These gloves protect hands and arms from ultra-cold materials and liquid nitrogen exposure. Sold as a pair and available in small, medium, large and extra large sizes.

6905 PORTABLE CRYOGENIC DEWAR*

Cryogenic Dewar designed for storing and dispensing small amounts of liquid nitrogen. Capacity of 25 litres.

6906 SHORT CRYOGENIC TRANSFER HOSE**

4 ft. (1.2 m) long flexible stainless steel hose suitable for transferring cryogenic fluids such as liquid nitrogen.

6907 LONG CRYOGENIC TRANSFER HOSE**

6 ft. (1.8 m) long flexible stainless steel hose suitable for transferring cryogenic fluids such as liquid nitrogen.

54006 DIFFUSER
Used for manual filling of LN using 6906/6907 transfer hoses.

**We recommend a 100-200 L LN tank for the 6875 or 6875D Freezer/Mills. Call your local LN supplier for details.*

***Transfer hoses and Autofill Systems are for use with low pressure (20-22 psi) LN tanks.*

BEFORE & AFTER


BEFORE


AFTER

PLASTIC TOYS

Cut toy into 1 cm pieces. 10 minute pre-cool period, 3 cycles of 3 minutes each, two minute cool down period with an impactor rate of 15 cps. In the 6875 Freezer/Mill.


BEFORE


AFTER

POTATOES

Cut into 1 cm pieces. Insert into 6801 vial in 6875 Freezer/Mill. 10 minute pre-cool, 3 cycles at 1 minute each with an impactor rate of 8 cps.


BEFORE


AFTER

BEEF LUNG

Samples were cut into small (5-8 mm) pieces, placed in a plastic bag then frozen. Frozen pieces were added to lightly pre-chilled 6801 vial, then placed in 6875 Freezer/Mill programmed for 12 min. Pre-cool, 3 cycles of 2 minutes at a rate of 12 cps.

BEFORE & AFTER


BEFORE


AFTER

DOG TREATS

20 grams of treats cut into 1-cm pieces. 15 pieces were loaded into a 6801 vial then pre-cooled in the 6875 Freezer/Mill for a 15 minutes. Then ground for 3 cycles of 2 minutes at a rate of 12 cps.


BEFORE


AFTER

POLYETHYLENE BEADS

15 grams of this sample was added into 6801 vial then pre-cooled in the 6875 Freezer/Mill for 10 minutes. Then ground for three 2 minute cycles at a rate of 15 cps.


BEFORE

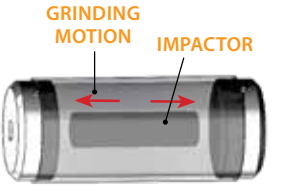

AFTER

HAIR

0.6 grams of trimmed hair was added into a 6801 vial then pre-cooled in the 6775 Freezer/Mill for 15 minutes. Then ground for three 2 minute cycles at a rate of 15 cps.

TECHNICAL INFORMATION

KEY POINTS ABOUT CRYOGENIC GRINDING



Cryogenic grinding has long been a vital sample preparation tool for the analytical chemist. The identification of the remains of Czar Nicholas II of Russia not only solved a mystery nearly eighty years old but also emphasized the importance of cryogenic techniques in both forensic and archaeological research. DNA was successfully extracted from the Romanov bone fragments after they had been ground in a SPEX SamplePrep Freezer/Mill.

Many analytical samples which are too flexible or sensitive to be impact-ground at room temperature can be embrittled by chilling, and then pulverized. These include polymers, rubber, textiles, cereal grains, hair, fingernails, skin, bone, and muscle tissue. There are also many samples which degrade in various ways during normal grinding, but whose critical properties are preserved by chilling. Coal, for example, can be cryogenically ground to retain its more volatile components, and clay minerals may be pulverized for XRD study without distorting their crystal structure. Bone, fingernails, and other biological materials can be cryogenically ground in preparation for nucleic acid extraction without damaging DNA and even RNA through heating.

LIMITS OF CRYOGENIC GRINDING

Not every substance that is difficult to grind at room temperature can be pulverized by cryogenic milling. SPEX SamplePrep recommends that you discuss your samples and grinding requirements with one of our specialists before selecting a mill. If there is any question about whether a sample can be ground in a Freezer/Mill we will request that a portion be submitted for test-grinding. Nearly every naturally occurring material of biological origin can be cryogenically ground, usually to a fine particle size. This includes hair, muscle tissue, bone, wood, plant stems and roots, seeds, cotton and fiber.

Polymers generally can be ground, but their physical form is important. Many flexible plastics which can routinely be milled in pellet form are difficult to grind when in the form of thin fibers or films. These remain flexible even at -200 °C. For the same reason,

polymers generally cannot be ground to as fine a particle size as biological samples. As the particles become smaller they become more flexible, and progressively more difficult to grind. Many silicone compounds remain elastic and hard-to-grind in any form.

The behavior of metals and metal alloys during cryogenic milling is highly variable. For example, impure copper as well as many copper alloys can be successfully ground, while pure copper remains malleable. Metal samples as a rule should be test-ground.

When a sample initially resists grinding, it may not be because it is "ungrindable." Many such samples respond to the strategies of reducing the sample volume, reducing the size of pieces, increasing grinding time, or lengthening the pre-cooling period up to 30 minutes. Improvements can also be made by using "filler" grinding agents such as quartz sand.

GUIDE TO FREEZER/MILL® SAMPLE CAPACITY

The optimum volume, weight, grinding time and impact frequency for any sample ground in the Freezer/Mill are determined by experimentation, the experience of the operator, and the requirements of the analyst. In most cases, the smaller the sample and the longer the grinding time, the finer the particle size will be. In cryogenic grinding the temperature also can affect the outcome; as a rule, the colder the sample, the finer it can be ground.

As an example of sample capacity, the 6751 Small Vial Set has a sample volume limit of about 5 mL and bulkier samples may restrict movement of the impactor. For efficient grinding, typical sample volume should be 1 to 2 mL, corresponding to weights of 1 to 2 grams for polymers and 2 to 4 grams for bone. Specialized grinding vials with similar capacity are the 6761 Poly-Vial for metal-free grinding of delicate samples, the 6771 low-chrome vial for grinding electronic components without adding chromium and the 6871S Stainless Steel Vial for grinding samples incompatible with polycarbonate.

The 6757 Microvial Set includes three microvials. Each Microvial can hold a small amount of sample, typically between 30 mg and 200 mg. The 6801 Vial has at least 50 mL maximum sample capacity, 10 times that of the 6751 Small Vial Set. Typical sample volumes are 10 to 20 mL, equivalent to weights of 10 to 20 grams for polymers and 20 to 40 grams for bone. The series of mid-size vials have about half the capacity of the 6801 vial and include the 6883 Mid-Size Low-Chrome vial, the 6885 Mid-Size Poly-Vial and the standard 6881 Mid-Size Vial.

CRYOGENIC GRINDING FOR ROHS/WEEE

The Restriction of Hazardous Substances (RoHS) and Waste Electrical and Electronic Equipment (WEEE) Directives of the European Union were introduced to minimize the accumulation of hazardous waste in landfills from the disposal of electrical and electronic equipment. The concentrations of hazardous substances such as lead, cadmium, mercury, chromium VI, polybrominated diphenylethers (PBDEs), polybrominated biphenyls (PBBs), Bis(2-ethylhexyl) phthalate (DEHP), Butyl benzyl phthalate (BBP), Dibutyl phthalate (DBP) and Diisobutyl phthalate (DIBP) are restricted in electrical and electronic products and/or components.

RoHS/WEEE states that if the component can be mechanically separated, then each component is subject to the RoHS limits. In order to get an accurate analytical result, these products and components must be reduced to homogeneous, representative samples. Many components such as circuit boards, wire, solder, polymers and resins are difficult if not impossible to grind using traditional methods. Cryogenic grinding in the Freezer/Mill® is the easiest way to homogenize these materials.

Consider analyzing circuit boards: for example, USB flash drives that act as portable hard disk drives, and the larger RAM modules that are installed on computer motherboards. These samples (Fig. 1) are composites of many materials: metals, resins, substrates, etc. A small piece taken from a board here or there at random would not

FIG. 1

BEFORE

FIG. 2

AFTER

be representative of the whole board, much less an entire batch of boards. Hence, sampling many boards, and in effect homogenizing them, assures that the sample used for analysis is representative of the batch. However, memory boards are made to be rugged and durable over a wide range of conditions, and are very difficult to grind into powder. Furthermore, some of the components may be more fragile than others. Metals, resins and other materials all have

distinct and different properties when put through conventional laboratory mills. Some components may grind at room temperature, and some may not. Some may require a shearing action and others abrasion.

When all of these components are part of the same integral memory board, it is impractical to break down the board into its several components, each suited for its own special type of mill. For such situations, cryogenic grinding in the Freezer/Mill is the answer. The general principle behind cryogenic grinding is to chill samples until they are brittle, and then break them up through impact, crushing, or shearing. In the case of the Freezer/Mill, the sample, such as the USB flash drive or RAM module, would be first cut into manageable pieces. These are then placed in the appropriately sized SPEX SamplePrep Low-Chrome Grinding Vial (6771, 6871, or 6883) with its magnetically driven Low-Chrome impactor. The vial is then loaded into the Freezer/Mill and immersed in liquid nitrogen until the contents are thoroughly chilled, usually in 10-15 minutes. The sample inside the vial is pulverized, and because the sample is isolated in a closed grinding vial, cross-sample contamination is easily controlled and sample integrity is maintained.

Grinding time can vary greatly depending on the type of sample and its pliability but a grinding time of 8-10 minutes can be expected in the case of the USB flash drive and RAM module. The result is fine, uniform powder (Fig. 2) can then be used for XRF, fusion, or other types of analyses.

USING THE FREEZER/MILL® FOR CRYOGENIC GRINDING OF PLANT AND ANIMAL TISSUE

Cryogenic grinding of plant and animal tissue is a very effective technique for the microbiologist. Samples that require extraction of RNA or proteins must be kept at -80° C or lower during the entire extraction process. Utilizing SPEX SamplePrep Freezer/Mills will maintain the sample temperatures well below these critical temperatures during the automated grinding sequence. For samples that are soft or flexible at room temperature, cryogenic grinding may be the only viable technique for processing your samples.

GRINDING TESTS WITH THE 6775 FREEZER/MILL®

MATERIAL	FORM	WEIGHT (G)	TIME*	FINAL SIZE MESH
Aluminum Foil	2 mil Piece	1[1]	3 x 2	100-200
Black loaded thermoplastic	Pellets	3	3 x 1	100-200
Candle Wax	Chunk	1.5	2	100-200
Cardboard	Corrugated	0.5	2 x 2	200
Fish Scales	10 mm Flakes	1.5	2 x 2	200
Hair	Dog Hair Clipping	0.5	2	200
Hot Melt Adhesive	Chunk	0.5	2 x 2	50
Human Tooth	Whole Tooth	2	1 x 2	200
Human Vertebrae	7 mm Pieces	2	1 x 2	200
Mouse Skin	Raw, ½ Animal	2[2]	3	200
Nylon[3]	3 mm Beads	2	2 x 2[4]	100-200
Permalloy[5]	2 mm Shot	2	3	30
Polyethylene	10 mil Sheet	1	2	200
Polypropylene	Fibers	1.5	3 x 2	100-200
Potatoes	Chunks	20g	3 x 3	200
Rubber	½ in. Thick Sheet	0.5	2 x 2	50-100
Rubber, Oil-Extended	5 mm Shearings	1.5[5]	2	25-50
Sheep Wool	Wad	0.5	2	200
Space Food	Stick	2	2	100
Teflon	2 mil Tape	3	2 x 2	100
Duct Tape	Tape	0.5	2 x 2	100
Urethane Elastomer	Pellets	1	3 x 1	100-200
Wood	Chips	1.3	2 x 2	100

**3 x 2 denotes three grinding periods of 2 minutes each with re-cooling of approximately 4 minutes between grinds.*

1. *0.5 g of Tide detergent was added.*
2. *Equal weight of sodium sulfate was added as dehydrating agent.*
3. *Three different nylons yielded similar results.*
4. *2x – two-minute grinds with one-minute cooling period between.*
5. *Equal amount of sand added; purpose: ethanol-toluene extraction.*

** results may vary with different models*

100 mesh = 149 microns

200 mesh = 74 microns

APPLICATION NOTES

The following application note excerpt are for the Freezer/Mill. To access our full library and download the complete application note visit www.spexsampleprep.com/appnotes.

APPLICATION NOTE SP002: GRINDING/HOMOGENIZATION APPLICATION: DOMESTIC WASTE

PREPARATION OF DOMESTIC WASTE FOR ELEMENTAL ANALYSIS

The Freezer/Mill (a cryogenic mill) is effective where other grinding processes fail. Flexible materials like plastics and biological samples embrittle at liquid nitrogen temperatures. The sample is hermetically sealed in the grinding vial and immersed in liquid nitrogen, so cross contamination is eliminated. As the Freezer/Mill is driven magnetically there is no wear and tear on moving parts.

APPLICATION NOTE SP005: DNA/RNA EXTRACTION / APPLICATION: PCR

RNA EXTRACTION FROM CARTILAGE TISSUE USING CRYOGENIC GRINDING

The object of the study was to determine if there is a connection between the seriousness of arthritic diseases, especially of the knee joint and certain genes. Total
RNA was isolated from cartilage tissue and the activity of certain genes was analyzed
via PCR or micro array analysis. With the multi-vial adapter option up to four samples can be homogenized simultaneously in the large Freezer/Mill®. By utilizing the precool function of the Freezer/Mill, the samples were held at about -196 °C for another two minutes. This assured sufficient cooling and therefore optimum brittleness.

APPLICATION NOTE SP006: MILLING/BLENDING (HOMOGENIZING) APPLICATION: PLANT PARTS

PREPARATION OF RICE HUSKS FOR THE ANALYSIS OF THEIR CONTENTS

The most important aspect for a successful grinding procedure is the sufficient cooling of the milled material to ensure brittleness of the sample. To this end the filled vial is inserted into the opening of the coil assembly and immersed into a liquid nitrogen bath. For brittleness the sample was left in the nitrogen bath for approximately 12 minutes.

APPLICATION NOTE SP008: STRUCTURAL CHANGES IN POLYMERS APPLICATION: POLYMER BLENDING

STRUCTURAL CHANGES IN POLYMERS BY MEANS OF CRYOGENIC GRINDING

Trials to introduce permanent changes in polymers by means of Cryogenic Mechanical Milling (CMM) with the Freezer/Mill® were started with two polymers; isotactic polypropylene (iPP) and syndiotactic polystyrol (sPS). After melting and re-crystallization, both cryogenically ground (at –196 °C) polymers showed characteristics that differ from the original polymers. DSC (Differential Scanning Calorimetry) tests showed changed crystallization temperatures, which indicates reduced molecular weight or a change in molecular weight distribution. The longer the samples were subjected to cryogenic grinding the bigger the change.

APPLICATION NOTE SP009: REDUCTION/HOMOGENIZATION APPLICATION: POLYMERS

NAFION®-POWDER FOR THE PRODUCTION OF FUEL CELL ELECTRODES

The DLR has now developed a method to apply the Nafion in powder form. For the production of this Nafion powder, the Freezer/Mill is utilized. As a polymer Nafion is very flexible and soft which obviously makes milling difficult. The Nafion granules (5 mm particle size) are reduced to approx. 10µm during grinding. The resulting powder is mixed with a catalyst and sprayed dry onto to the 25-175 µm thick membrane.

HOW ARE YOU PREPARING YOUR CANNABIS SAMPLES FOR TESTING?

SPEX CertiPrep offers Certified Reference Materials (CRMs) for all of the common contaminants found in medicinal and recreational Cannabis, including pesticide residues, residual solvents, terpenes, among others. All of our CRMs come backed with our ISO 17034, ISO/IEC 17025 and ISO 9001 certifications and accreditations, guaranteeing the product you purchase is measured to the highest industry standards available. We continually update our product offering to meet the changing Cannabis testing regulations.

Visit www.cannstandards.com to learn more.

Standards Include:

- Pesticide Residues
- DEA Controlled Substances
- Residual Solvents
- Heavy Metals
- Terpenes
- Pyrethroids
- Chlorinated Hydrocarbons
- Organophosphates
- Carbamates
- Custom mixes to your exact specification also available

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HIGH-ENERGY BALL MILLS

The **8000-series Mixer/Mills®** are laboratory mills that pulverize small amounts of hard, brittle samples to analytical fineness. They are functionally described as shaker mills or high-energy ball mills. The vial, which contains a sample and one or more balls, 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 clamp's motion develops strong G-forces in the vial, to pulverize the toughest rocks, slags and ceramics.

APPLICATIONS

Blends Powders
Makes Emulsions
Mechanical Alloying
Nanomilling
Extractions
Slurry Grinding
XRF
Mechanochemistry

SAMPLE TYPES

Rocks
Minerals
Sand
Cement
Slag
Ceramics
Glass
Pharmaceuticals
Animal & Plant Tissue
Seeds
Catalyst Supports

8000M MIXER/MILL®

The 8000M Mixer/Mill® is a high-energy ball mill that accommodates samples up to 20 g for grinding, and up to 60 mL for blending powders or mixing emulsions. Accepts a range of regular grinding/mixing vials from different materials and smaller vials with a proper adapter.

Steel housing and rugged construction ensures a long life of heavy work.

Timer is factory-set for 100-minute range. Optional chip is available to extend timer range to 175 hours (10,500 minutes).

Equipped with safety interlock system for operator protection.

Forced air cooling fan.

Clamp swings back-and-forth with a short lateral "figure-8" movement.

Shock-mounted electric motor for tabletop operation.

SPECIFICATIONS

VOLTAGE	100–120 V, 60 / 50 Hz 200–240 V, 50 / 60 Hz
DIMENSIONS	21 in. (53 cm) x 22 in. (56 cm) x 14 in. (36 cm)
WEIGHT (LBS)	77 lbs. (35 kg)
MOTOR	1/3 hp, 1725 RPM @ 60 Hz 1425 RPM @ 50 Hz
CLAMP MOVEMENT	2 1/3 in. (5.9 cm) back-and-forth, 1 in. (2.5 cm) side-to-side
CLAMP SPEED	1,060 cycles/minute 115 V, 60 Hz or 875 cycles/minute 230 V, 50 Hz. Alternate pulley supplied with 230 V model for 1060 cpm operation.
POWER CORD	Supplied with a 3-prong grounded cord 115 V, 60 Hz, or a 2-prong grounded European cord for 230 V, 50 Hz.

MIXING & GRINDING OPTIONS

- One large vial
- Four 3/4" (19-1 mm) vials
- Seven 1/2" (12-7 mm) vials

8000D MIXER/MILL®

The 8000D Mixer/Mill® is an efficient two-clamp laboratory mill that offers increased sample throughput while using the same vials as the 8000M. Grinds up 40 grams of sample per run, 20 grams per clamp.

Steel housing and rugged construction ensures a long life of heavy work.

Timer is factory-set for 100-minute range. Optional chip is available to extend timer range to 175 hours (10,500 minutes).

Forced air cooling fan.

Equipped with safety interlock system for operator protection.

Clamps swing back-and-forth with a short lateral "figure-8" movement.

Shock-mounted electric motor for tabletop operation.

SPECIFICATIONS

VOLTAGE	100–120 V, 60 / 50 Hz 200–240 V, 50 / 60 Hz
DIMENSIONS	25 in. (64 cm) x 21 in. (54 cm) x 14 in. (36 cm)
WEIGHT (LBS)	93 lbs. (42 kg)
MOTOR	1/3 hp, 1,725 RPM @ 60 Hz 1,425 RPM @ 50 Hz
CLAMP MOVEMENT	2 1/3 in (5.9 cm) back-and-forth, 1 in (2.5 cm) side-to-side.
CLAMP SPEED	1060 cycles/minute (115 V) or 875 cycles/minute (230 V)
POWER CORD	Supplied with a 3-prong grounded cord 115 V, 60 Hz, or a 2-prong grounded European cord for 230 V, 50 Hz.

MIXING & GRINDING OPTIONS

- One large vial per clamp
- Four 3/4" (19.1 mm) vials per clamp
- Seven 1/2" (12.7 mm) vials per clamp

SMALL VIALS & VIAL SETS

For use with adapters in the 8000M and 8000D Mixer/Mills.

3111 POLYSTYRENE VIAL

2.5 mL grinding vial that includes a slip-on polyethylene cap. Vial is 1/2 in. diameter x 1 in. long (12.7 x 25.4 mm). Sold as a bag of 100 vials.

3114 STAINLESS STEEL VIAL SET

2.5 mL grinding vial. 1/2 in. diameter x 1 in. long (12.7 x 25.4 mm). Includes a slip-on cap and one 1/4 in. (6.35 mm) stainless steel ball.

3116 POLYSTYRENE VIAL

5 mL grinding vial with slip-on polystyrene cap.Vial is 1/2 in. diameter x 2 in. long (12.7 mm x 50.8 mm). Sold as a bag of 100.

3116PC POLYCARBONATE VIAL

5 mL grinding vial with slip-on polyethylene cap. 1/2 in. diameter x 2 in. long (12.7 mm x 50.8 mm). Sold as a bag of 100.

3117 HARDENED TOOL STEEL VIAL SET

2.5 mL grinding vial set. 1/2 in. diameter x 1 in. long (12.7 mm 25.4 mm). Includes a slip-on cap and one ¼ in. (6.35 mm) steel ball.

3120 AGATE VIAL SET

3.5 mL grinding vial set. 7/8 in. (22.2 mm) diameter x 2 in. (50.8 mm) long. Includes one 1/4 in. 3118A agate ball, can be used for wet or dry grinding.

**3111, 3116 and 3116PC accept 3112 3/8 in. (9.5 mm) acrylic balls.*

3127 HARDENED TOOL STEEL VIAL SET

5 mL vial set. 3/4 in. diam. x 17/8 in. long (19.1 mm x 47.6 mm). Includes a center cylinder, 2 slip-on caps, and one 1/4 in. (6.35 mm) steel ball.

6133 POLYPROPYLENE VIAL

12 mL polypropylene grinding vial with flip-top cap. Vial is 3/4 in. diameter x 2 in. long (19.1mm x 50.8 mm). Use 3112 or 8006A balls. Sold as a bag of 100 vials.

6133PC-T POLYCARBONATE VIAL

12 mL polycarbonate grinding vial that includes a slip-on polyethylene cap. Vial is 3/4 in. diameter x 2 in. long (19.1mm x 50.8 mm). Use 3112 or 8006A balls. Sold as a bag of 100 vials.

5004 TUNGSTEN CARBIDE-LINED VIAL SET

5 mL grinding vial set. 3/4 in. x 2 1/8 in. (19.1 mm x 54 mm). 2 Delrin® caps with tungsten carbide inserts, 6 disposable methacrylate center cylinders and two 5/16 in. (7.9 mm) tungsten carbide grinding balls.

6134 POLYPROPYLENE VIAL

35 mL vial with attached cap,1.19 in. diameter x 3 in. long (3.02 cm x 7.62 cm). Suitable for slurry grinding. Sold as a bag of 100 vials.

6135 POLYPROPYLENE VIAL

75 mL vial with attached cap. Vial is 1.57 in. diameter x 2.99 in. long (3.99 cm x 7.59 cm). Sold as a bag of 100 vials.

LARGE VIALS & VIAL SETS

For use in 8000-series Mixer/Mills

8001 HARDENED STEEL VIAL SET

65 mL vial set. 2 ¼ in. diam. x 3 in. long (5.7 cm x 7.62 cm). Includes a screw-on cap with O-ring, two ½ in (12.7 mm) and four ¼ in.(6.35 mm) steel balls.

8002 POLYSTYRENE VIAL

135 mL vial with plastic screw-on cap., 2 1/8 in. diameter x 2 ½ in. long (5.4 cm x 6.7 cm). Sold as bag of 100 vials. Can be used with 3112 and 8006A Methacrylate Balls.

8003 ALUMINA CERAMIC VIAL SET

45 mL vial set. 2 ¼ in. diam. x 2 ¾ in. long (5.7 cm x 7.0 cm). Includes 99.5% alumina ceramic grinding vial, 2 slip-on alumina ceramic caps, one ½ in. (12.7 mm) alumina ceramic ball and 8 corprene gaskets.

8015 CLAMP FOR 8003 VIAL SET

This clamp permits slurry grinding in the 8003 Alumina Ceramic Grinding Vial Set. Sold in pairs.

8004 TUNGSTEN CARBIDE VIAL SET

55 mL vial set, 2 ¼ in. diam. x 2 ½ in. long (5.7 cm x 6.35 cm). Tungsten carbide-lined body, 2 tungsten carbide-lined aluminum caps, two 7/16 in. (11.2 mm) tungsten carbide balls and 8 corprene gaskets.

8004SS STAINLES STEEL-JACKETED TUNGSTEN CARBIDE VIAL SET

55 mL vial set, 2 ¼ in. diam. x 2 ½ in. long (5.7 cm x 6.35 cm). Tungsten carbide lined stainless steel body, two tungsten carbide lined stainless steel caps, two 7/16 in. (11.2 mm) tungsten carbide balls and 8 Viton Gaskets.

8005 ZIRCONIA CERAMIC VIAL SET

45 mL vial set. 2 ½ in. diameter x 2 in. long (6.35 cm x 6.8 cm). Includes two ½ in. zirconia ceramic balls and seven corprene gaskets.

8007 STAINLESS STEEL VIAL SET

65 mL vial set, 2 1/4 in. (5.7 cm) diameter x 3 in. (7.62 cm) long. Includes screw-on cap, O-ring, two 1/2 in. (12.7 mm) and four 1/4 in. (6.35 mm) stainless steel balls.

8008 SILICON NITRIDE VIAL SET

45 mL vial set, 2 ½ in. diameter x 2 11/16 in. long (6.35 cm x 6.8 cm). Solid silicon nitride vial and slip-on cap, two 8008A Silicon Nitride Balls, ½ in. (12.7 mm) and seven corprene gaskets.

8009 ROUND-ENDED HARDENED STEEL VIAL SET

35 mL vial set, 2 3/8 in. diameter x 3 in. long (6.0 cm x 7.62 cm). 1 screw-on cap, one O-ring, and one 1 in. (25.4 mm) steel ball.

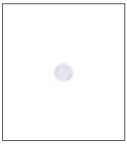
8014 AGATE VIAL SET

45 mL vial set, 2 ¼ in. diam.x 2 ¾ in. long (4.7 cm x 7.0 cm). Agate vial body, 2 slip-on agate caps, two 8014A agate balls, ½ in. (12.7 mm) and eight corprene gaskets.

8020 POLYCARBONATE GRINDING JAR, 75ML

75 mL Polycarbonate jar with methacrylate balls. For use on the 8000M and 8000D Mixer/Mill. Jars are sold individually. Suitable for slury grinding up to 40 mL. This vial can also be used with the 8007B Stainless Steel ball set.

GRINDING MEDIA



3112 METHACRYLATE BALLS

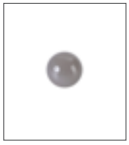
3/8 in. (9.5 mm) diameter. Fits all SPEX SamplePrep plastic vials. Sold in units of 100.

3114SB STAINLESS STEEL BALL SET

1/4 in. (6.35 mm) diameter stainless steel (440C) ball; for the 3114 vial set. Four balls per set.

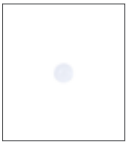
3117B HARDENED TOOL STEEL BALL SET

1/4 in. (6.35 mm) inch diameter steel ball; for 3117 and 3127 Vial Sets. Four balls per set.



3118A AGATE BALL

1/4 in. (6.35 mm) diameter agate ball; for 3120 Vial Set.



3119 METHACRYLATE BALLS

1/8 in. (3.2 mm) diameter. Fit all SPEX SamplePrep plastic vials. Sold in units of 100.

5004A TUNGSTEN CARBIDE BALL SET

5/16 in. (7.9 mm) diameter tungsten carbide ball; for 5004 Vial Set. Four balls per set.

8001B STEEL BALL SET

Two 1/2 in. (12.7 mm) and four 1/4 in. (6.35 mm) hardened steel balls for 8001 and 8009 vial sets.

8003A ALUMINA CERAMIC BALL

1/2 in. (12.7 mm) diameter alumina ceramic ball for 8003 Vial Set.

8004A TUNGSTEN CARBIDE BALL

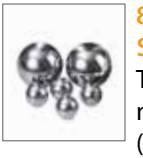
7/16 in. (11.2 mm) diameter tungsten carbide ball for 8004 and 8004SS Vial Sets.

8005A ZIRCONIA CERAMIC BALL

1⁄2 in. (12.7 mm) diameter zirconia ceramic ball for 8005 Vial Set.

8006A METHACRYLATE BALLS

1/2 in. (12.7 mm) diameter. Fit 6133, 6134, 6135, 8002, and 8006 vials. Sold in units of 100.



8007B STAINLESS STEEL BALL SET

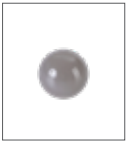
Two 1/2 in. (12.7 mm) and four 1/4 in. (6.35 mm) hardened 440C stainless steel balls for 8007 vial set.

8008A SILICON NITRIDE BALL

1/2 in. (12.7 mm) diameter silicon nitride ball for 8008 vial set.

8009B STAINLESS STEEL BALL

1 in. (25.4 mm) steel ball or 8009 vial set.



8014A AGATE BALL

1/2 in. (12.7 mm) diameter agate ball for 8014 vial set.

VIAL ADAPTERS

Special adapters enable simultaneous running of multiple samples in the 8000 Series Mixer/Mills. The 8010 and 8011 multiple-sample adapters accommodate 1/2 in. (12.7 mm) and 3/4 in. (19.1 mm) diameter vials, respectively.

8010 MULTIPLE-SAMPLE ADAPTER

Holds seven vials ½ in. (12.7 mm) in diameter. Must be used with 8012 Adapter to run vials 1 in. (25.4 mm) long in 8000D Mixer/Mill.

8011 MULTIPLE-SAMPLE ADAPTER

Holds four vials ¾ in. (19.1 mm) in diameter. Must be used with 8012 Adapter with to run 3127 vials in 8000D.

8012 VIAL CLAMP ADAPTER FOR THE 8000D MIXER/MILL

Used with 8010 adapter with vials 1 in. (25.4 mm) in length (3111, 3114, 3117) or when running 3127 vials with 8011 adapter in the 8000D.

8016 ADAPTER FOR 6134 VIAL

Aluminum adapter for 6134 vial. For use with the 8000M or 8000D.

8017 ADAPTER FOR 6135 VIAL

Aluminum adapter for 6135 vial. For use with the 8000M or 8000D.

8018 ADAPTER TO HOLD SEVEN 2 ML VIALS

Aluminum multi vial adapter for seven 2mL plastic vials for Mixer/Mill.

8019 ADAPTER TO HOLD SEVEN 5 ML VIALS

Aluminum multi vial adapter for seven 5mL plastic vials for Mixer/Mill

STANDARD/OPTIONAL GASKETS AND O-RINGS FOR 8000 SERIES MIXER/MILL® VIALS- EXPLANATION OF MATERIALS USED

Viton® - A fluoroelastomer with good resistance to oils and most other fluids. Chemicals known to degrade Viton are ketones, ethers, esters, amines, strong bases (NaOH) and acetic acid. Incompatible with Vertrel® XF. (Part no. 51746 standard O-ring for 8001, 8007, 8009; Part no. 39322 standard gasket for 8004SS; Part no. 39322 optional gasket for 8004; Part no. 39515 optional gasket for 8003, 8005, 8008, 8014).

Corprene - A combination of cork and neoprene with good resistance to oils, solvents, and most other fluids. Chemicals to avoid are organic solvents, strong bases, and strong acids. Incompatible with Vertrel® XF. (Part no. 10009 standard gasket for 8003, 8005, 8008, 8014; Part no. 10010 standard gasket for 8004 ; Part no. 10010 optional gasket for 8004SS).

EPDM - Ethylene propylene diene monomer with good resistance to phosphate esters, ketones, alcohols, and most other fluids. Chemicals to avoid are aromatic hydrocarbons, fuel oils and strong acids. Recommended for use with Vertrel® XF. (Part no. 40002 optional gasket for 8004, 8004SS; Part no. 40004 optional gasket for 8003, 8005, 8008, 8014; Part no. 51715 optional O-ring for 8001, 8007, 8009).

Teflon® - Synthetic fluoropolymer with excellent resistance to a wide range of chemicals. (Part no. 51714 optional O-ring for 8001, 8007, 8009 vials.)

TECHNICAL INFORMATION

GRINDING VIAL SAMPLE SPECIFICATIONS

P/N	GRINDING SAMPLE VOLUME	TYPICAL GRINDING SAMPLE WEIGHT	BLENDING SAMPLE VOLUME	SUITABLE FOR SLURRY GRINDING OR BLENDING
HARDENED STEEL				
3117	0.2-0.6 mL	0.5 g	1 mL	No
3127	0.5-1.0 mL	1 g	2 mL	No
8001	30 mL	10 g	25 mL	Yes
8009	20 mL	10 g	25 mL	Yes
STAINLESS STEEL				
3114	0.2-0.6 mL	0.5 g	1 mL	No
8007	30 mL	10 g	25 mL	Yes
TUNGSTEN CARBIDE				
5004	0.5-1.5 mL	1 g	2.5 mL	Yes
8004	3-10 mL	10 g	25 mL	Yes
8004SS	3-10 mL	10 g	25 mL	Yes
ALUMINA CERAMIC				
8003	2-8 mL	10 g	20 mL	Yes
AGATE				
3120	0.2-0.6 mL	0.5 g	1 mL	No
8014	2-8 mL	10 g	20 mL	No
ZIRCONIA CERAMIC				
8005	2-8 mL	10 g	20 mL	Yes
SILICON NITRIDE				
8008	2-8 mL	10 g	20 mL	Yes
PLASTIC				
3111	-	-	1 mL	Yes
3116	-	-	2 mL	Yes
3116PC	-	-	2 mL	Yes
6133	-	-	5 mL	Yes
6133PC	-	-	5 mL	Yes
6134	-	-	15 mL	Yes
6135	-	-	40 mL	Yes
8002	20-50 mL	-	50 mL	Yes
8006	3 – 10 mL	-	20 mL	Yes

MECHANICAL ALLOYING

Mechanical alloying, also referred to as reactive milling, is a process originally developed for the production of oxide dispersion strengthened superalloys. Today, mechanical alloying is often used as a solid-state powder processing technique that generates powders with unique microstructures. A high-energy ball mill can be used to accomplish this. Over the past few decades, the 8000M Mixer/Mill, widely known as the "SPEX Mill", has become the industry standard for mechanical alloying applications. Benefits of this mill include the high energy of the milling action, continuous forced air cooling and the durability of the motor which allow running for extended periods.

The 8000M Mixer/Mill is equipped with a timer that is factory set for a 100 minute time range. However, mechanical alloying requires significantly longer grinding times. For these applications, we offer an optional chip to extend the timer range to 10,500 minutes. This chip is available as either a factory-installed or user-installed option. Due to additional wear that can occur on the Mixer/Mill from extended running times, installation of this chip changes the warranty terms and a routine schedule of preventative maintenance is strongly suggested. For additional information please contact our SPEX SamplePrep Application Specialist.

Hundreds of articles have been published in peer reviewed scientific journals regarding the Mixer/Mill and its use for mechanical alloying. This includes mechanical alloying techniques, evaluations of grinding vial materials, and numerous other topics. The following publication list is intended to highlight some key publications and is not intended to be comprehensive. If you are considering the Mixer/Mill for your own mechanical alloying application, we encourage you to do your own search for application publications and references.

Temperature of the milling balls in shaker and planetary mills. Takacs, L., McHenry, J. S. Journal of Materials Science, Vol. 41, Issue: 16, August 2006. pp. 5246 – 5249.

Modeling of comminution processes in Spex Mixer/Mill. Concas, A., Lai, N., Pisu, M., Cao, G. Chemical Engineering Science, Vol. 61, Issue: 11, June, 2006. pp. 3746-3760.

A study of mechanical alloying processes using reactive milling and discrete element modeling. Ward, T.S., Chen, W., Schoenitz, M., Dave, R.N., Dreizin, E.L. Acta Materialia, Vol. 53, Issue: 10, June, 2005. pp. 2909-2918.

Comparative study of SPEX and planetary milling methods for the fabrication of complex metallic alloy nanoparticles. A Zolriasatein, A Shokuhfar, F Safari, N Abdi - Micro & Nano Letters, 2018 – IET, Volume 13, Issue 4, April 2018, p. 448 – 451

Self-ignited synthesis of nanocomposite powders induced by Spex mills; modeling and optimizing. Reza Ebrahimi-Kahrizsangia, Majid Abdellahia, Maryam Bahmanpour. Ceramics International, Volume 41, Issue 2, Part B, March 2015, Pages 3137-3151

Ball Milled Si-W Alloys: Part I. Microstructural and Phase Evolution during Ball Milling. Yijia Liu, Benjamin Scott, M. N. Obrovac. J. Electrochem. Soc. 2019 volume 166, issue 6, A1170-A1175

BEFORE & AFTER

SUPERALLOY

Ground in the 8000M Mixer/Mill for 15 mins. Result was a slightly lumpy powder. It was then ground in the ShatterBox for 6 mins. The result was a fine, almost silky, texture.


BEFORE


AFTER

GRANITE

11 grams of granite was ground in the 8000M Mixer/Mill for 18 minutes in an 8001 steel vial with 2 steel balls.


BEFORE


AFTER

RED SANDSTONE

11 grams of red sandstone was ground in the 8000M Mixer/Mill for 18 minutes in an 8001 steel vial with 2 steel balls.


BEFORE


AFTER

GRINDING TESTS WITH 8000-SERIES MIXER/MILLS

MATERIAL	FORM	VIAL - METHOD	TIME (MIN)	AMOUNT (G)	% PASSING 325 MESH (45 MICRONS)
Antimony	Pieces	L-D	5	26	97
Asbestos	fluff	WC-D	10	*	*
Bauxite	60 mesh	HS-W	30	3	*
Bismuth	Chunks	PJ-D	20	5	75
Bone	Chunks	AC-D	10	*	35
Boron Carbide	Chunks	WC-D	15	7	100
Brake Linings	Chunks	WC-D	*	*	*
Carbon (activated)	Pieces	HS-D	10	10	90
Carbnauba 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	Pieces	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	HS-E	30	3	100
Porcelain	Chunk	WC-D	15	6	83
Potassium Pyrosulfate	Fused Button	PV-D	10	5	100
Reforming Catalyst	3mm Beads	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	6mm Lumps	L-D	10	5	30
Slag (blast furnace)	-	HS-W	20	3	100
Slag (copper)	100 mesh	WC-W	10	5	84
Slag (open hearth)	-	HS-W	20	3	100
Straw	-	HS-D	10	5	**
Tomato Stems	-	HS-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

Table References:
HS – 8001 Hardened Steel Vial | L – 8006 Acrylic Vial | WC – 8004 Tungsten Carbide Vial | W – Wet ground (slurry) | PJ – 8002 Polystyrene Vial | D – Dry ground | AC – 8003 Ceramic Vial | PV – 6133 Polystyrene Vial
**- Suitable for X-ray or optical emission spectroscopy | **- Satisfactory for extractions*

PROPERTIES OF GRINDING CONTAINER MATERIALS

This chart reviews the key properties of each grinding container material, including major and minor elements, hardness, resistance to abrasion, durability, and efficiency. These are usually the basis for selecting the proper container for a particular application.

MATERIAL	MAJOR ELEMENTS	MINOR ELEMENT(S)	HARDNESS	RESISTANCE TO ABRASION	DURABILITY	COMPARATIVE EFFICIENCY
Hardened Steel	Fe	Cr, Si, Mn, C	Mohs: 5½ - 6 Rockwell:C 60-65	Moderate	High	High
Stainless Steel	Fe, Cr	Ni, Mn, S, Si[1]	Mohs: 5 - 5½ Rockwell:C 55-60	Moderate	High	High
Low-chrome Steel	Fe	C, Mn, Si, Mo	Mohs: 5 - 5½ Rockwell:C 55-60	Moderate	High	High
Tungsten Carbide	W,C,Co	Ta, Ti, Nb	Mohs: 8½ + Knoop: 1400-1800	High	Long-wearing, subject to breakage	Very High
Alumina Ceramic	Al	Si, Ca, Mg	Mohs: 9 Rockwell: R45N 74-79 Knoop 1160	Very High	Long-wearing, brittle	Moderate
Agate	Si	Al, Na, Fe, K, Ca, Mg[1]	Mohs: 6 - 7 Knoop: 550-800	Extremely High	Very long-wearing	Moderate
Zirconia	Zr	Y,Mg,Hf	Mohs: 8½ Rock-well:R45N 74-79 Knoop: 1160	Extremely High	Very long-wearing	Moderate
Silicon Nitride	Si	Y, Al, Fe, Ca	Mohs: 8½ + Knoop 1600	Extremely High	Very long-wearing	Moderate
Plastic	C	-	Mohs: 1 ½	Low	Low, disposable	Low for grinding; High for blending

[1]*All reported less than 0.02%*

APPLICATION NOTES

The following are excerpts of application notes for the Mixer/Mill. To access our full library and download the complete application note visit www.spexsampleprep.com/appnotes.

SP003: NANOTECHNOLOGY / APPLICATION: PRODUCTION OF NANO POWDERS

PRODUCTION OF NANO-CRYSTALLINE OXIDIZED CERAMICS WITH HIGH-ENERGY BALL MILLING

Analyzing particle size effects in nanocrystalline materials requires a technique in which one can adjust the particle size. In this study various nano-crystalline materials were produced using a ball mill (8000M Mixer/Mill®, SPEX SamplePrep; equipped with alumina and zirconia vials). Ball milling is particularly suitable for this task as it is easy to use and allows the grinding of a relatively large amount of material as well as a large variety of different materials.

The analyzed media were: Li_2O, $LiNbO_3$, $LiBO_2$, B_2O_3, TiO_2 and Li_2O:B_2O_3 mixtures. The average particle size was determined by the grinding time and subsequently analyzed by means of X-ray Diffraction (XRD) and Transmission Electron Microscopy (TEM). The lithium containing materials were selected because of their potential use as solid electrolytes. TiO_2 is interesting with regard to its use as a photo catalyst. For hygroscopic materials the corundum grinding vial was filled in an argon atmosphere and put into an airtight stainless steel container.

HOW-TO GUIDELINE: MILLING & EXTRACTION / APPLICATION: CRUDE FAT

FEEDSTUFFS ANALYSIS – CRUDE FAT (HEXANE EXTRACTABLES)

Lipids are recovered by simultaneous milling and extraction with hexane in a sealed ball mill vial. The method is applicable to feedstuffs, corn germ and other components derived from the milling of corn. Grind a 50-100 g sample to a uniform particle size (10 mesh) in a grinder or cutting mill.

Mix the sample thoroughly and weigh accurately about 4-5 g into the steel grinding vial. Add one 12mm and two 6 mm steel balls and 25 mL of n-hexane. Seal the vial and mill in the Mixer/Mill for 15 mins. Place a filter paper in the funnel and filter the milled sample slurry into a clean and dry vacuum flask. Quantitatively transfer the residual oil from the steel milling vial and cap with the aid of three 10 mL portions of hexane, and wash residual oil from the sample with another three 10 mL portions of hexane. Transfer the filtered hexane and oil miscella into a dried and tared 150 mL beaker. Rinse the vacuum flask residue into the beaker with small portions of hexane.

Evaporate the solvent on a steam-heated water bath under the hood. Place the beaker with residue in the air oven and dry it 1 hour at 100 °C. Remove the beaker and cool in a desiccator. Weigh the beaker and calculate the dry extract weight.

RING & PUCK MILLS

The **ShatterBox®** is a ring & puck mill that accommodates sample sizes ranging from 2 to 150 grams. Ideal for pulverizing dry, brittle samples and slurry grinding. It swings a dish-shaped grinding container, with a puck and (typically) a ring inside, in a tight high-speed circle. The sample is quickly crushed between the wall and floor of the container and the moving puck/ring grinding elements. Smaller grinding containers and those made with ceramics have a puck-shaped grinding element; larger containers have a ring and puck. Grinding containers are fitted with a gasket to prevent sample loss during grinding. The ShatterBox holds one large or three small grinding containers.

APPLICATIONS

Rapid dry or wet grinding to analytical fineness of hard, brittle samples.

SAMPLE TYPES

Pharmaceuticals

Ores

Minerals

Rocks

Slag

Ceramics

Dried Plant

Soil

SHATTERBOX®

The 8530 ShatterBox® is a ring & puck mill with sound-proof enclosure. Accommodates sample sizes ranging from 2 to 150 grams. Ideal for pulverizing dry, brittle samples and slurry grinding.

Programmable, 10-minute adjustable timer with LCD display and push-button membrane switch.

Lid locks down when mill is running. Soundproof enclosure reduces noise levels.

Lockable casters enable easy movement of the mill.

Swings a dish-shaped grinding container in a tight high-speed circle.

Cam-action lever requires only moderate hand pressure to secure the grinding dish in the ShatterBox and is adjustable for containers of different heights.

SPECIFICATIONS

VOLTAGE	100–120 V, 60 / 50 Hz 200–240 V, 50 / 60 Hz
DIMENSIONS	19 in (48 cm) x 19 in (48 cm) x 40 in (102 cm)
MOTOR	1⁄2 hp @ 835 RPM
WEIGHT (LBS)	207 lbs. (94 kg)
POWER CORD	Supplied with a 3-prong grounded cord 115 V, 60 Hz, or a 2-prong grounded European cord for 230 V, 50 Hz.

MIXING & GRINDING OPTIONS

One large or three small grinding containers.

ACCESSORIES

GRINDING CONTAINERS

8501 HARDENED STEEL GRINDING CONTAINER

Includes puck, ring, dish, O-ring gasket and lid. 6.9 in. diam. x 2.9 in. high, 17.5 lbs. (17.5 cm x 7.5 cm, 8 kg). Grinding load is 20-50 mL. Volume 270 mL.

8504 TUNGSTEN CARBIDE GRINDING CONTAINER

Includes puck, ring, dish, O-ring gasket and lid. 6.9 in. diam.x 3.2 in. high, 29 lbs. (17.5 cm x 8 cm, 12.5 kg). Grinding load 20-60 mL. Volume 240 mL.

8505 ALUMINA CERAMIC GRINDING CONTAINER

Includes puck, dish with O-ring gasket and lid. 6.3 in. diam. x 3.2 in. high, 11 lbs. (15.2 cm x 8 cm, 5 kg). Grinding load 15-40 mL. Volume 170 mL.

8506 ZIRCONIA CERAMIC GRINDING CONTAINER

Includes puck, dish with O-ring gasket and lid. 6 in. diam. x 3.2 in. high, 13 lbs. (15.2 cm x 8 cm, 6 kg). Grinding load 15-40 mL. Volume 170 mL.

8507 SMALL HARDENED STEEL GRINDING CONTAINER

Includes puck, dish with gasket, and lid. 3.6 in. diam. x 2.3 in high, 4 lbs. (9 cm x 5.4 cm, 1.9 kg). Volume 90 mL. Grinding load 5-20 mL. Used with 8507R rack.

8508 SMALL TUNGSTEN CARBIDE GRINDING CONTAINER

Includes puck, dish and lid. 3.6 in. diam. x 2.3 in. high, 7.7 lbs. (9.2 cm x 5.9 cm, 3.5 kg). Grinding load 5-20 mL. Volume 80 mL. Used with 8507R rack.

8521 LARGE HARDENED STEEL GRINDING CONTAINER

Includes puck, ring, dish with O-ring gasket and lid. 8 in. diameter x 3.3 in. high, 29 lbs. (20.3 cm x 8.2 cm, 13 kg). Volume 650 mL. Grinding load 30-100 mL.

PREP-AID® GRINDING AID

3650 PREP-AID VERTREL® XF

Liquid fluorocarbon grinding aid. Prevents caking, reduces contamination, evaporates after grinding. One-quart bottle.

RACKS AND HOLDERS

8507R RACK

Holds one or three 8507 or 8508 grinding containers in ShatterBox. Rack base plate has handles to lift Rack into or out of ShatterBox, and four locating pins for positioning one or three grinding containers. Rack lid is matched to ShatterBox clamp, and holds grinding containers down during operation. When 8507R Rack is used with three grinding containers, all three should be the same kind, either 8507 or 8508.

TECHNICAL INFORMATION

GRINDING CONTAINER SPECIFICATIONS

PART NUMBER	MATERIAL	SAMPLE VOLUME	TYPICAL SAMPLE	SUITABLE FOR BLENDING	SUITABLE FOR SLURRY GRINDING
8501	Hardened Steel	20-50 mL	75 g	Yes	Yes
8507	Hardened Steel	5-20 mL	15 g	Yes	Yes
8521	Hardened Steel	30-100 mL	150 g	Yes	Yes
8504	Tungsten Carbide	20-60 mL	75 g	Yes	Yes
8508	Tungsten Carbide	5-20 mL	15 g	Yes	Yes
8505	Alumina Ceramic	15-40 mL	40 g	Yes	Yes
8506	Zirconia Ceramic	15-40 mL	40 g	Yes	Yes

GRINDING TESTS WITH THE 8501 GRINDING CONTAINERS

MATERIAL	FORM AS RECEIVED	TIME (MIN)	AMOUNT (G)	% PASSING 325 MESH
Asbestos	Fiborous	12	20	100
Cement (Portland raw mix)	>60 mesh	2.5	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
Fiberglass	Thin sheets	2	10	100
Fluorite	>100 mesh	3	50	100
Garnet (synthetic)	Chunks	2	200	15
Glass	Chunks	6	35	100
Graphite Mineral Fiber	Fiber	2	15	100
Insulation	Fiber	4	15	100
Oil Shale	6.4mm	3	6	100
Pesticide	<100 mesh	15	50	100
Phosphate, raw mix	<60 mesh	2.5	40	100
Iron Powder	<80 mesh	6	5	68
Sand	<10 mesh	10	100	100
Slag, blast furnace	Chunks	1	10**	100

GRINDING TESTS WITH THE 8504 GRINDING CONTAINERS

MATERIAL	FORM AS RECEIVED	TIME (MIN)	AMOUNT (G)	% PASSING 325 MESH
Asbestos	Fiborous	-	-	-
Cement (Portland raw mix)	>60 mesh	-	-	-
Ferro-chromium	>100 mesh	-	-	-
Ferro-manganese	>200 mesh	-	-	-
Ferro-molybdenum	<80 mesh	-	-	-
Ferro-niobium	<80 mesh	-	-	-
Ferro-silicon	<80 mesh	-	-	-
Ferro-titanium	<80 mesh	-	-	-
Fiberglass	Thin sheets	2	15	100
Fluorite	>100 mesh	-	-	-
Garnet (synthetic)	Chunks	10	200	96
Glass	Chunks	2	35	100
Graphite Mineral Fiber	Fiber	-	-	-
Insulation	Fiber	-	-	-
Oil Shale	6.4mm	-	-	-
Pesticide	<100 mesh	-	-	-
Phosphate, raw mix	<60 mesh	-	-	-
Iron Powder	<80 mesh	-	-	-
Sand	<10 mesh	4	100	100
Slag, blast furnace	Chunks	-	-	-

**Sodium alkylarylsulfonate added, 5%*
***Household detergent (Tide) added, 10%*

BEFORE & AFTER SAMPLES

GLASS


BEFORE


AFTER

GARNET-BEARING ROCK


BEFORE


AFTER

SANDSTONE


BEFORE


AFTER

ZINC ORE


BEFORE


AFTER

SHATTERBOX® TECHNICAL INFORMATION

Since its introduction over sixty years ago, the SPEX SamplePrep ShatterBox has become one of the most popular "swing mills" in the United States. It is the most efficient way to pulverize up to 150 grams of brittle material to analytical fineness.

APPLICATIONS

The ShatterBox typically grinds cement mix, rocks, slags, soils, ceramics, and ores, but has been used for hundreds of other materials including sulfur pellets, dried marsh-grass, and pharmaceuticals. The original SPEX SamplePrep ShatterBox, was developed for cement plants and steel mills with open, noisy lab conditions and is still used in many locations where the working environment requires a rugged, simple mill. The 8530 ShatterBox is ideal for labs concerned with minimizing noise and maximizing safety.

GRINDING CONTAINERS

The standard grinding containers, with a nominal volume of about 100 mL, are available in hardened steel (8501), tungsten carbide (8504), alumina ceramic (8505), and zirconia ceramic (8506). Typical grinding loads for samples such as cement, rock, or slag are 50 grams for the steel and tungsten carbide containers and 30 grams for the ceramic containers. Note that the actual capacity of the containers is much higher, and (for example) the steel container has been used for as little as 4 grams and as much as 100 grams. However, grinding efficiency decreases as the sample becomes more tightly packed.

There are also small grinding containers made of hardened steel (8507) and tungsten carbide (8508), which can be run either one or three at a time with the 8507R Rack. These grinding containers have about a fifth the capacity of the standard grinding containers and a typical sample size of 10 grams. An oversize steel grinding container (8521) is also available and has approximately a 150 mL capacity. This corresponds to a typical sample size of 75-100 grams.

GRINDING RESULTS

Grinding times are usually between two and five minutes, with resultant particle size well below 200 mesh (approximately 50 microns). With grinding aids, smaller samples can be reduced below 10 microns.

KEY POINTS ABOUT GRINDING

GRINDING CONTAINER MATERIALS

The selection of appropriate grinding and mixing containers is important. The proper grinding vial or dish will greatly enhance analytical accuracy. The grinding container is generally harder than the material to be ground and should be of a substance whose presence in the sample will not interfere with analysis. SPEX SamplePrep's containers have been selected with care to offer optimum capability in the lab.

Methacrylate and polystyrene vials and balls are ideal for pulverizing soft, brittle materials and for mixing and storing powders. With these vials only traces of organic impurities are added to your sample.

Hardened steel is durable and tough, suitable for general-purpose grinding; it's the workhorse of the lab. When hard substances are ground in steel containers, some Fe and Cr contamination can be expected. Stainless steel is less subject to chemical attack, but contributes Ni as well. Low-chrome steel rusts easily but can be used to grind samples for RoHS/WEEE testing. Halide-releasing compounds corrode steel and should be ground in tungsten carbide, alumina, agate, zirconia, or silicon nitride containers.

Tungsten carbide vials and dishes are the most effective and versatile of all. Tungsten carbide is substantially harder and heavier than steel, grinding is faster and contamination is minimal. Other than tungsten, cobalt (a binder) is the major contaminant. If cared for, tungsten carbide containers will last indefinitely. They're recommended as a long-term "best buy" for grinding almost anything.

Alumina ceramic is ideal for extremely hard samples or in cases where steel and tungsten carbide contaminants are objectionable. All SPEX SamplePrep alumina components are 99.5% pure aluminum oxide, with some silicon, calcium, and magnesium present. It is lightweight and brittle but very abrasion-resistant. Necessary for an important minority of samples, alumina ceramic vials are a helpful addition to any lab's grinding armory.

Agate is harder than steel, and chemically inert to almost anything except HF. It's also brittle and must be handled with care. Agate vials are for the grinding and mixing of samples when organic and metallic contamination is equally undesirable. Agate is 99.9% silica and is extremely wear-resistant.

Silicon nitride is a tough space-age material with remarkable wear characteristics, and hardness superior to agate and zirconia. If it is important to have a container whose only major contaminant is silicon, consider SPEX SamplePrep silicon nitride. It is extremely durable compared to agate, and while it contains some yttria and alumina, overall contamination levels will be very low.

Zirconia is a ceramic which in many ways approaches the ideal grinding medium. Since it is both hard and tough it wears very slowly, adding little contamination. It is about one and one-half times as dense as alumina, and grinds yttrium almost as fast as steel, because it is mostly zirconium oxide with low percentages of magnesium oxide and hafnium oxide, the contamination SPEX SamplePrep zirconia ceramic does contribute is often not important to the analyst.

CONTAMINATION

Processing a sample always contaminates it. Successful analysis depends on recognizing the sources of contamination and controlling them. When the contaminants are known and can be quantified, analytical results can be refined accordingly.

As the grinding container is the major source of contamination, its selection is critical. In general, one's objective should be to minimize contamination levels while avoiding elements which will interfere with analysis. An example is the grinding of steel slags in a tungsten carbide container: tungsten carbide grinds rapidly, and the expected low-level contaminants of tungsten, carbon, and cobalt are not generally looked for in these slags. SPEX SamplePrep's selection of grinding container materials gives you maximum flexibility in choosing the best approach for your samples and analytical aims.

Major, minor, and trace elements predictably found in SPEX SamplePrep grinding containers are listed in this book. However, strictly speaking, almost no two grinding containers will have exactly the same elemental profile. There are many different steels, carbides, and ceramics, each with specific compositions. Often the formulas are proprietary, so that a type of tungsten carbide engineered to have specific properties will have a different makeup from two different manufacturers. In addition, there are inevitable variations from batch to batch of the same material, both in the exact proportions of the major elements and in trace element composition.

Because of these variations in grinding container composition, we strongly recommend determining the exact elemental profile of your individual grinding containers, preferably with your own analytical equipment and techniques. The simplest approach is to grind samples of known composition and see what is added by grinding. Lacking known samples, one may grind portions of a single sample for increasing lengths of time, and check to see which elements increase in proportion to grinding time. Once the contributed impurities and their proportions are known for a grinding container, the resulting profile can be fitted to the analytical results, regardless of the actual contamination level. (While this level is important, it clearly will vary with the composition and condition of the grinding container, the size, hardness, and toughness of the sample, and grinding time).

GRINDING AIDS

When samples agglomerate or "cake" during grinding, further particle size reduction is clearly inhibited. Caking can result from moisture, heat, static charge accumulation, the fusing of particles under pressure, and other causes. Many of the techniques are devoted to getting around caking.

Slurry grinding is an obvious approach; if particles remain in suspension during grinding, they are unlikely to cake. Water, alcohol, or other liquids are added to the sample before grinding, and removed afterwards. Although slurry grinding is a reasonably reliable way of grinding a sample to micron-sized particles, it is sloppy and time-consuming, requires a leak-proof grinding container, and adds extra steps to one's sample preparation procedure.

Dry grinding is simpler and quicker, but requires much more careful matching of the technique to the sample. If the caking is due to moisture, as in many soils and cements, the sample can be dried before grinding. Other samples can be successfully ground with a variety of additives. Dry soaps/detergents are lubricants, and some also include an abrasive; graphite is an anti-static agent as well as a lubricant; there are many proprietary grinding aids as well, which may contain an abrasive, a lubricant and a binding agent. Other grinding aids include polyvinyl alcohol, phenyl acetate and aspirin.

The use of propylene glycol (one drop for up to ten grams of sample, roughly 0.3 wt.%) for laboratory fine grinding of Portland cement and many minerals is suggested. In a swing mill such as the SPEX SamplePrep ShatterBox, oven-dried samples can be ground quickly

to less than ten microns without agglomeration or sticking to the mill walls. Propylene glycol must be used safely, after consulting the material safety data sheet.

Vertrel® XF, a DuPont product sold as a cleaning fluid, is finding increased acceptance as a grinding aid. A fluorocarbon fluid, it prevents sample caking during grinding, and quickly evaporates from an open grinding container without leaving any residue. Our experience is that the "grindability" of almost any sample is enhanced by the use of Vertrel XF, contamination is lowered, and the grinding container is easier to clean. Typical XRF samples such as cement, rock, clinker, and similar material can be routinely ground below 10 microns. Vertrel XF also lowers contamination levels from the grinding container, and is available as a Prep-Aid product.

The following technique is suggested for SPEX SamplePrep equipment: in an 8501 Hardened Steel Grinding Container load together 10 grams of sample, 2.5 grams of 3642 Cellulose or 3644 Ultrabind binder, and 7 mL of Vertrel XF. Grind in an 8530 ShatterBox for 2.0 minutes, open the grinding container in a hood until the Vertrel XF has evaporated. Prepare a 3614 40mm Pellet Die with a flared 3617 Spec-Cap, and transfer the ground sample/binder powder to the die. Press at 20 tons for 0.3 minutes in an X-Press.

CLEANING CONTAINERS

Grinding containers should be cleaned between sample runs to avoid cross contamination, and the procedure can be as simple or as complex as your analytical objectives warrant. In some applications a simple wipe-down with ethanol may suffice; another practical approach is to brush out a container, then briefly grind an expendable portion of the next sample and discard it. Washing a container with detergent and warm water is often effective.

For more thorough cleaning one may grind one or more batches of pure quartz sand, and then wash the container thoroughly. In extreme cases, such as the plating of container walls with a malleable metal, chemical cleaning or multiple grinds with quartz may be necessary. An effective single-step grinding procedure for most grinding containers is to grind pure quartz sand together with hot water and detergent, then rinse and dry the container. Drying is speeded by the use of a blow-dryer or similar appliance. A safety advantage of this cleaning method is that it controls respirable airborne dust.

A cleaning procedure is easily evaluated by grinding and analyzing a known sample, or even by checking the impurities appearing in

successive batches of ground quartz sand. It should be noted that grinding containers become more difficult to clean with age because of progressive pitting and scratching of the grinding surfaces.

Hardened steel and even stainless steel containers can rust. While iron oxide coatings can be removed by warm dilute oxalic acid solution or abrasive cleaning, we recommend that steel containers be thoroughly dried after cleaning and, if stored, kept in a plastic bag with a desiccating agent.

RESPIRATORY PROTECTION

The general objective of sample grinding is to convert an inhomogeneous solid to a fine, homogeneous powder. Inevitably, some of this powder is released into the environment, usually during the emptying or cleaning of the grinding container. We strongly recommend the wearing of an approved dust-mask during this procedure, and suggest the use of a laboratory fume hood. Even harmless rock and cement samples can become potentially harmful to the respiratory system when finely pulverized.

INFRARED MULLS

KBr pellets and Nujol mulls are quickly prepared with SPEX SamplePrep Mixer/Mills using small plastic (3111), agate (3120) and steel vials (3114). The polystyrene spectrum appears as a constant background and can easily be subtracted. Steel and agate produce no IR background.

WHEN TO PULVERIZE

The non-homogeneity of materials in the real world presents a serious sampling dilemma: how to represent a large, often nonuniform whole with a small analytical sample. Often the best approach is first to take a quantity of the material large enough to be compositionally representative and reduce it to a fine homogeneous powder. Then the sample can be adapted to a particular analytical technique, and used as is, pressed into a sample disk, fused, dissolved, etc. SPEX SamplePrep has laboratory mills and grinding containers capable of pulverizing and blending all kinds of samples, from metals to plastics, rocks to living tissue, and pesticides to pharmaceuticals.

HOW TO SELECT A LABORATORY MILL AND GRINDING CONTAINER

SamplePrep mills are intended for the analytical laboratory. They are not for batch or production milling, but for rapid, efficient grinding of analytical samples ranging in amounts from less than one gram up to approximately 100 grams. The SPEX SamplePrep Freezer/Mills, Mixer/Mills and ShatterBox all have separate grinding containers for individual samples, so after use the container is cleaned and not the mill.

If your samples can be pulverized by impact at room temperature, you have a choice of several proven SPEX SamplePrep mills. The ShatterBox is a ring-and-puck mill that is ideal for rapid grinding of samples up to approximately 150 grams, in 5 minutes or less. The 8000M and 8000D Mixer/Mills are high-energy ball mills that not only pulverize samples in the 10 gram range but are suitable for blending powders and making emulsions up to approximately 60 mL. All these mills have multiple-sample capacity with smaller samples, and a wide choice of grinding container sizes and materials, for maximum on-the-job flexibility over a wide range of applications.

The SPEX SamplePrep Freezer/Mills are unique cryogenic mills that can grind almost any "ungrindable" sample, including all plant and animal tissue, most polymers, and countless other samples that are resistant to room-temperature milling. Freezer/Mills require liquid nitrogen for chilling the sample, and maintaining cryogenic temperatures in the vial during grinding.

The following chart shows the composition of Standard Steels used in SPEX SamplePrep Equipment and Accessories. To discuss any of these mills and containers, and their suitability for your applications, feel free to contact our product specialists. We also offer test-grinding of your samples in our applications lab, or we can loan you a mill for short-term trials in your own lab.

The recommended maximum sample volume for grinding is approximately 20% of a container's volume, for blending 40–50%. Optimum grinding efficiency is achieved with samples toward the middle of the volume range. The "typical grinding sample" is quartz sand, with a density of approximately 1.3g/mL, in a volume conducive to efficient grinding. The analyst must choose the proper weight for their sample, based on its density and other characteristics.

Chart B: Composition of Standard Steels Used in SPEX SamplePrep Equipment & Accessories

Compositions are theoretical, actual lot analyses are not available. The major element is iron, minor elements listed below will vary. Additional trace elements may be present and will change from lot to lot. Actual composition of individual grinding vials should be determined by analyzing known samples ground in those containers, and determining which element(s) were added during the sample grinding.

TYPE NUMBER	CARBON %	MANGANESE %	PHOSPHORUS %	SULFUR %	SILICON %	CHROMIUM %	NICKEL %	MOLYBDENUM %	ZIRCONIUM %	WHERE USED
303	0.15 max	2.00 max	0.20 max	0.15 min	1.00 max	17-19	8-10	0.60 max	0.60 max	3114
304	.08 max	2.00 max	0.045 max	0.030 max	1.00 max	18-20	8-12	-	-	8004SS
410	0.42	0.40	0.040 max	0.030 max	0.60	14.25	-	-	-	5200, 3127
440C	1.10	0.60	0.040	0.030	1.00	16-18	-	0.50	-	2150, 3114SB, 3127, 3610, 3613, 3614, 3616, 3623, 6751, 6757, 6781S, 6801, 6881, 8007, 8007B
52-100	0.95-1.1	0.20-0.35	0.03	0.03	-	1.3-1.6	-	-	-	3117B, 8001, 8009, 8501, 8507, 8521
ASTM 06	1.45	1.00	-	-	0.90	-	-	0.25	-	6771, 6871, 6883
Tool Steel	0.90	1.10	-	-	0.30	0.50	-	-	-	3117

PELLET PRESSES

Our range of compact bench-top hydraulic pellet presses are ideal for pressing sample pellets for XRF, IR, and other analytical methods. Typical pressing time is two minutes or less. Our automated press can be programmed for repetitive pressing for high-throughput labs. Our manual presses are an economic alternative for lower throughput labs. Our presses are designed to withstand the most demanding lab environments with extraordinary durability.

APPLICATIONS

Pressing sample pellets for XRF/IR

SAMPLE TYPES

Pharmaceuticals
Cement
Minerals
Rocks
Slag
Soil

3636 X-PRESS®

The 3636 X-Press® is a 35-ton (31.8 metric ton) laboratory press that is automated and programmable. It is ideal for repetitive pressing of sample pellets for XRF, IR, and other analytical methods. Typical pressing time is two minutes or less. The X-Press can also be operated manually.

Safety door and interlock with automatic pump shut-off safety valve.

Adjustment screw secures the loaded sample die placed on top of the platen.

Accepts all standard die sets.

Touch-screen display allows user to enter maximum pressure, hold time, and release time for automated runs, start and stop press, or select manual mode.

Handle and handwheel moves the adjustment screw to secure the sample.

Access panel can be removed to fill the oil reservoir or check the oil level.

Manual pressure relief valve used to release the hydraulic system pressure in an emergency or for system maintenance.

SPECIFICATIONS

VOLTAGE	100–120 V, 60 / 50 Hz 200–240 V, 50 / 60 Hz
DIMENSIONS	20.0 in. (50.8 cm) x 13.0 in. (33 cm) x 22.5 in. (57.2 cm)
MOTOR	1/3 hp
WEIGHT (LBS)	142 lbs. (65 kg)
PLATEN SIZE	3.25 inches diameter (8 cm)
PLATEN MOVEMENT	1.0 inch (2.5 cm)
FORCE	10–35 tons ram pressure (9.1-31.8 metric tons or 20,000 – 70,000 lbs.)
POWER CORD	Supplied with a 3-prong grounded cord 115 V, 60 Hz, or a 2-prong grounded European cord for 230 V, 50 Hz.

DIE SET OPTIONS

The X-Press accepts all standard die sets.

CARVER® MANUAL PRESS

These manual hydraulic laboratory pellet presses can be operated under the most primitive conditions. Economical alternative to motorized presses for labs with a small sample load. Rugged, reliable and noted for their extraordinary durability. Typical samples include: cement, rocks, minerals, soils, ceramics, pharmaceuticals.

3621 Manual/Press

PART NUMBER: 3621

Full-size 12-ton (10.9 metric ton) hydraulic laboratory pellet press that accepts 10 mm, 13 mm, 31 mm, 35 mm, and 40 mm pellet die sets. Manually controlled. Ideal for pressing sample pellets for XRF, IR, and other analytical techniques. CE compliant model available by request.

3622 Manual/Press

PART NUMBER: 3622

Full-size 25-ton (22.7 metric ton) hydraulic laboratory pellet press that accepts 10 mm, 13 mm, 31 mm, 35 mm, and 40 mm pellet die sets. Manually controlled. Ideal for pressing sample pellets for XRF, IR, and other analytical techniques. CE compliant model available by request.

3626 Manual/Press

PART NUMBER: 3626

Bench top 12-ton (10.9 metric ton) hydraulic laboratory pellet press that accepts 13 mm pellet die sets. Manually controlled. Ideal for pressing sample pellets for IR. CE compliant model available by request.

DIE SETS

3613 DIE SET – 13 MM

Includes evacuable die body, base, plunger, two 13 mm polished steel pellets, O-ring vacuum seals, knock-out ring for sample disk extraction. Load limit 10 tons (9.1 metric tons).

3623 DIE SET – 31 MM

Includes evacuable die body, base, plunger, two 31 mm polished steel pellets, O-ring vacuum seals, knock-out ring for sample disk extraction. Load limit 50 tons (45.4 metric tons).

3616 DIE SET – 35 MM

Includes evacuable die body, base, plunger, two 35 mm polished steel pellets, O-ring vacuum seals knock-out ring for sample disk extraction. Load limit 50 tons (45.4 metric tons).

3614 DIE SET – 40 MM

Includes evacuable die body, base, plunger, two 40 mm polished steel pellets, O-ring vacuum seals, knock-out ring for sample disk extraction. Load limit 50 tons (45.4 metric tons).

REPLACEMENT PELLETS

Replacement or spare steel pellets for die operation can be purchased separately. For routine pressing of abrasive materials, we offer 31 mm, 35 mm and 40 mm tungsten carbide pellets which can be substituted for polished steel pellets. Tungsten carbide pellets are much harder than steel, but more brittle, and are not guaranteed against breakage. All replacement pellets are sold in pairs.

PELLETS	DIE SET
3613ST Steel Pellets – 13 mm	For 3613 die, sold in pairs.
3623ST Steel Pellets – 31 mm	For 3623 die, sold in pairs.
3623C Tungsten Carbide Pellets - 31 mm	For 3623 die, sold in pairs.
3616ST Steel Pellets – 35 mm	For 3616 die, sold in pairs.
3616C Tungsten Carbide Pellets – 35 mm	For 3616 die, sold in pairs.
3614ST Steel Pellets – 40 mm	For 3614 die, sold in pairs.
3614C Tungsten Carbide Pellets – 40 mm	For 3614 die, sold in pairs.

SLEEVE AND PLUNGER SETS

Our Sleeve-and-Plunger sets allow easy preparation of XRF disks with binder matrix supporting a layer of sample. Makes sturdy disks, prevents damage to die; ideal for smaller amounts (<1 g) of analyte. These sets can be purchased separately or together with our dies at a discount.

3614W 40 MM DIE SLEEVE AND PLUNGER SET

Used with 3614, 40 mm Evacuable Pellet Die and Prep-Aid Sample Binder

3616W 35 MM DIE SLEEVE AND PLUNGER SET

Used with 3616, 35 mm Evacuable Pellet Die and Prep-Aid Sample Binder.

3623W 31MM DIE SLEEVE AND PLUNGER SET

Used with 3623, 31 mm Evacuable Pellet die and Prep-Aid Sample Binder.

SPEC-CAPS®

SPEX SamplePrep Spec-Caps® are shallow, thin-walled aluminum cups which are routinely used in the production of pressed powder sample disks for OES, XRF, and other analytical techniques. The Spec-Cap® forms the bottom and sides of the finished pellet. Thus reinforced, sample disks are resistant to chipping and breaking, and are more easily handled, marked, and stored than unclad disks.

PREPAID® BINDERS

SPEX PrepAid binders can be blended with sample at 10% by weight to form XRF sample disk, or used undiluted as sample matrix with Sleeve-and-Plunger Set. Will bond 200-250 disks or provide matrix for 25-35 supported sample disks.

See pages 119 and 120 for details about SPEC-Caps® and PrepAid Binders.

TECHNICAL INFORMATION

PRESSING & PELLETIZING

Analytical spectroscopic methods such as XRF, OES, and IR often require samples in the form of flat-surfaced disks. Although sample disks can be cast from a fusion melt, (refer to section on Fusion Products), they often begin as a powder and are pressed to shape in a pellet die.

The following how-to suggestions apply chiefly to XRF sample preparation, but the basic principles of forming sample disks are the same for OES, spark ablation, IR, etc. The major differences are the diameters of the disks, and the nature of the binders or additives. IR disks are 13 mm across and consist largely of pure potassium bromide (KBr) with the pulverized sample blended in; no Spec-Cap®-type jacket is used, as the sample disk must be able to transmit infrared light. OES and spark ablation systems generally use 31 mm disks. As the disk must be electrically conductive for these techniques, most samples are blended with 50% pelletizing-grade graphite, and often pressed in an aluminum Spec-Cap®.

The diameter of XRF sample disks is dependent on the size of the spectrometer sample holder, which may be 31 mm, 35 mm, 40 mm, or even larger. XRF disks do not require a binder or Spec-Cap® if the sample coheres under pressure, but most analysts use a binder or a Spec-Cap® or both. The prime requirement for an XRF binder is that it does not contribute impurities. XRF binders include cellulose, paraffin, graphite, orthoboric acid, polyvinyl alcohol, and proprietary products with special properties, e.g. Ultrabind®.

For preparation of pellet samples, SPEX SamplePrep offers a range of laboratory presses, pellet die sets, and die accessories. Accessories include: aluminum Spec-Caps® to form and protect pellets; cellulose and paraffin Prep-Aid binders and UltraBind®. Sleeve-and-Plunger sets to form sample pellets with a thin layer of sample on a binder matrix are also available.

SPEX SamplePrep dies and presses ensure production of uniform sample disks, whatever the sample or analytical technique.

PREPARING POWDER SAMPLES FOR XRF

There are many different procedures for preparing powdered samples for XRF analysis. Typically, however, a representative quantity of the sample is first pulverized, then split to obtain enough powder for an XRF sample disk, usually 6 to 10 grams. That powder is blended with a

binder if necessary, and placed in a pellet die (with or without a Spec-Cap®) to be pressed into a sample disk which will hold together and has a flat, compositionally uniform surface. This disk is then placed in the sample holder of the XRF spectrometer.

An alternate technique, particularly useful when only a small amount of sample is available, incorporates a thin layer of sample on a disk of binder. The SPEX SamplePrep Sleeve-and-Plunger set, used with the appropriate pellet die, makes this procedure easy.

The sampling method and the amount of sample to be ground, the type of mill, grinding time, size of die and what pressure to use, whether to include a binder or press the disk in a Spec-Cap® may vary and must be worked out to suit the samples and analytical requirements. Running a series of standards or known samples will help to confirm that your chosen procedure results in accurate, reproducible data.

PARTICLE SIZE EFFECTS

It is impossible to say a priori how fine to pulverize a given sample. It has been known for many years that the X-ray fluorescence intensity from a sample will increase as the particle size of the sample is decreased. This is due to the reduction in the size and extent of the voids in the sample. By the same reasoning, as the particle size of one of two sample components is decreased, it will yield a higher intensity relative to the component of fixed particle size. Further, if the particle size of both components is decreased, their respective intensities may increase or decrease depending on their relative absorption coefficients. Fortunately, when the particle size becomes small enough, the intensities stabilize.

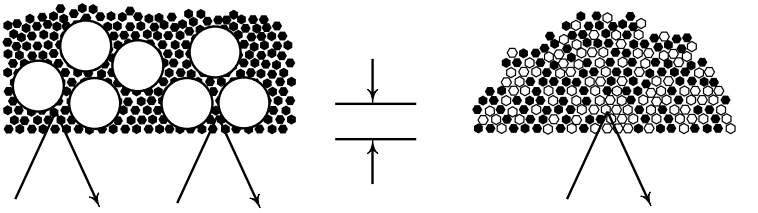
As an X-ray enters a pelletized sample disk the exciting radiation is absorbed by the matrix. When a particle within the matrix absorbs radiation, the resulting fluorescence is also absorbed, in whole or in part, by the matrix. Hence there is a limit to the depth to which the existing radiation can penetrate and result in fluorescence emitted from the sample. This depth is usually called the critical or infinite thickness.

As particle size becomes small relative to the critical thickness, fluorescence intensities emitted from different sample components approach stable values. This is shown graphically below. In case A, the average penetration of the X-ray is of the same order of magnitude as the large particles, and a change in the size of these particles would have a substantial effect on the fluorescent intensity. This results from the filling of the voids by components with smaller particle sizes. In

case B, since the average penetration depth covers many particles of many components, changing the particle size should have little or no effect.

In practice, the limiting particle size is often determined empirically by grinding the sample for a given length of time, measuring the particle size, analyzing the sample, then repeating at longer grinding times until the intensities reach a plateau.

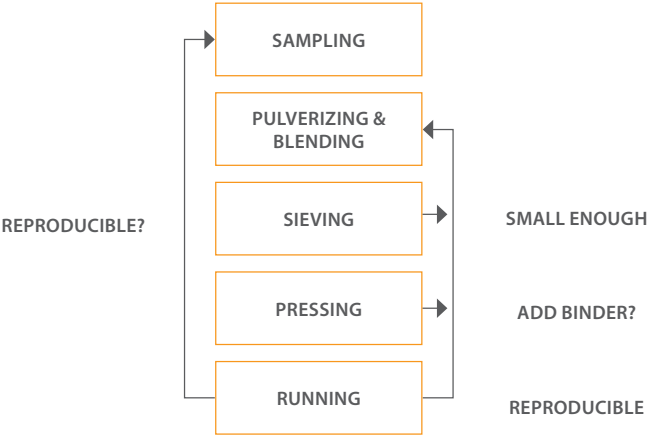
PRESSING AND PELLETIZING



A B

Critical Thickness

Particle size should be small, relative to the penetrationdepth of the X-rays.



The correct particle size is often determined empirically

TECHNIQUE FOR PRESSING A SAMPLE DISK

In pressing samples for XRF, the loaded pellet die is placed in a hydraulic press, and the pressure is raised to a level that will cause the sample or sample/binder mixture to cohere into a stable sample disk.

A basic pressing sequence consists of raising the pressure to a specific level, holding it there for a certain length of time, and then releasing it, preferably slowly.

The maximum pressure for a given task varies considerably, depending on the size of the die and the nature of the sample. Obviously maximum pressures should not exceed the load limits of the die. The 13 mm die has a 10-ton limit, so some care must be exercised, but 31 mm and larger dies usually have load limits higher than either the capacity of the press or the pressure required to form a sample disk. Typical pressures for a 31 mm disk are from 20 to 25 tons; for a 35 mm disk, from 22 to 30 tons; and for a 40 mm disk, from 25 to 35 tons. Some samples cohere adequately at low pressures, but uniform high pressure is recommended. As "infinite depth" is very shallow for XRF analysis of most elements, a matter of microns, compaction of the sample decreases pore space and increases analytical accuracy.

Holding time and bleed time are both important. If a sample is simply brought to maximum pressure, and the pressure is abruptly released, the sample disk often does not hold together. This may be due to elastic rebound of gases trapped in the sample, because a binder may take time to completely penetrate the sample, or for other reasons. A holding time at maximum pressure of at least 30 seconds is recommended. Some analysts hold pressure for 5 minutes or more. During holding time the pressure should be maintained as well as possible. An advantage of the Automated X-Press is that it turns on the pump if the pressure drops more than 1 ton during the holding period.

A gradual release of pressure after the hold period is perhaps even more important than prolonged holding time. A minimum bleed time of 15 seconds is recommended. For samples that do not bind well, several minutes may be appropriate. A slow, careful bleed period can be difficult to accomplish with manual presses, as the pressure release control is often not sensitive, but it is still never a good idea to dump pressure abruptly in any pelletizing procedure. Another significant advantage of the X-Press is that lengthy hold times and precisely controlled pressure release can be programmed in, and will remain the same, sample after sample. The overall uniformity of the sample disks (and hence of the analytical results) will inevitably be greater.

THE USE OF THE VACUUM IN PELLET PRESSING

All SPEX SamplePrep evacuable pellet dies have a vacuum hose attachment, which enables a vacuum pump to be hooked up to the die before and during a pressing operation. Most samples contain gases, moisture, and pore space, and removal or reduction of these can in fact affect the stability and uniformity of the sample disk. In the case of 13 mm dies, which are primarily used to produce KBr disks for infrared spectrometry, the use of a vacuum is necessary to draw moisture out of the KBr. Most XRF analysts do not bother to evacuate their 31 mm, 35 mm or 40 mm dies during pressing, but in fact the withdrawal of air and moisture from the sample can improve disk compaction and quality. Troublesome samples will often benefit from this technique. When evacuating a die, it is advisable to make sure both the upper and lower O-rings are in place and in good condition.

SET-UP AND LOADING OF THE SPEX SAMPLEPREP EVACUABLE PELLET DIE

Most evacuable pellet dies operate in the same way, by pressing the analytical sample between two polished pellets of steel or tungsten carbide. The simplest way of loading the die is to assemble the die barrel and die base; insert the lower polished pellet, polished side up, into the bore; pour the sample into the bore, level it, and add the upper polished pellet, polished side down; then insert the plunger, and place the assembled die in the press. This approach works well if the sample coheres well under pressure and is neither abrasive nor adhesive. Once the sample disk is pressed, it should be fairly easy to remove it from the die, either by hand or with the use of the knock-out ring supplied with each SPEX SamplePrep die.

When the sample does not hold together well after pressing, or sticks to the die or scratches it, there are various pelletizing aids available. These include Spec-Caps®, Prep-Aid binders, Sleeve-and-Plunger sets, and various anti-sticking agents.

CARE AND MAINTENANCE OF THE SPEX SAMPLEPREP EVACUABLE PELLET DIE

The SPEX SamplePrep evacuable pellet die is a precision tool which must be handled carefully and diligently maintained for proper operation. Although in design and function such dies are very simple, precise fit of the working parts is absolutely necessary, and easily jeopardized. A pellet die in good condition will produce thousands of sample disks without difficulty; a damaged or heavily worn die is likely to produce frustration, delays, and chipped or broken sample

disks. Any damage to the polished pellets or the bore of a pellet die should be corrected immediately.

In a pellet die in proper condition, the polished pellets should pass smoothly through the die bore without binding, but their fit should be so precise that the pulverized sample will not "leak" around the pellet edges. A good test is to assemble the die bore and base, seal the evacuation port with a fingertip, and place a polished pellet (polished side up) in the bore. It should remain at or near the top of the bore, and spring back when pushed down lightly, due to compression of the air inside the die. When the evacuation port is unsealed, the pellet should drop smoothly to the bottom of the bore. If the polished pellet sinks immediately with the evacuation port sealed, it is either a loose fit, or the seal between the die body and base is damaged.

Polished pellets are made with close tolerances and sharp edges so that a sample powder will not "feather" into the gap between the pellet and the bore. (When this happens, the sample disk may have a raised, crumbly lip and the disk and upper polished pellet may be difficult to remove from the die). The edges of these pellets are by far the most vulnerable part of a die, as they can be dented or chipped by dropping them even a short distance onto a hard surface. Such dents are extremely dangerous to the integrity of the die. Not only can they cause the pellets to bind in the die, but also, under the high pressures of pelletizing, the stressed edge of the pellet can spall off. The resulting chip can be dragged through the bore, scarring it deeply, and potentially jamming the plunger and ruining the die.

Minor damage to the polished pellets and die bore should be immediately corrected with a fine-grained (e.g. 600 grit) emery paper. If after this the pellet will still pass smoothly through the die bore, and its leading edge is not significantly chipped, the pellet and die can continue in service. (A badly chipped pellet should be retired, as sample can wedge into the space left by the chip, making it difficult to extract the pellet from the die. In addition, further chipping is likely to occur under pressure). A lightly scarred die bore, properly smoothed, can continue in use.

Damage to the polished face of the pellet should also be avoided, but will probably not affect the functioning of the die. Analytical accuracy is what suffers. Scratches on the order of 20–30 microns can cause shielding effects in the sample disk, and overall abrasion of the polished pellet face can very slightly change the geometry and distance in the critical relationship between X-ray tube, sample disk, and detector. Obviously if analytical results are being distorted

because of the condition of the polished pellets, it is time to replace them, but the degree to which such distortion is tolerable will vary considerably from user to user. A simple way to check polished pellets is to press two sample disks of identical material, one with a pristine polished pellet and the other with the worn pellet, and compare the analytical data.

In handling the pellet die, some simple rules should be kept in mind: keep the die clean, and always treat it as the precision tool it is. Pellet dies should be cleaned after every use, to avoid both sample cross-contamination and the possibility of disk jamming or sticking from sample build-up. In cleaning the polished pellets, treat them like glass; in other words, use the same cleaning technique you would for a glass lens or mirror. Steel has a hardness similar to glass, and it is important to avoid scratching the polished surface.

Remember that the polished pellets are the most critical parts of the die, and the most easily damaged. When inserting the polished pellets into the bore of the die, take extra care that they do not jam; the fit is so precise that a very slight tilt will cause them to stick. When this happens, free the pellet gently. Above all, do not push it down further and make the situation worse, as this can cause the pellet to chip, and quite possibly ruin the die. A simple technique for inserting the polished pellet into the die bore is to hold the trailing edge of the pellet lightly with the finger-tips, and rotate it gently in the mouth of the bore to make sure it is properly lined up. When a polished pellet is placed in the die bore, it should move freely. If it does not, careful corrective action should be taken immediately.

PROPER USE OF THE SPEX SAMPLEPREP SPEC-CAP®

Spec-Caps® are shallow aluminum cups in which a sample is pressed. A sample disk properly prepared with a Spec-Cap® will be encased by the Spec-Cap® on one side and around its edge, allowing the disk to be written on and handled without crumbling or contamination. The Spec-Cap® also protects the bore of the die from abrasive samples, which with time can enlarge or damage the die, and the paint on the 3615, 3617, and 3619 Spec-Caps® acts as a lubricant, making it easier to remove the sample disk from the die.

There are two types of Spec-Caps®, unpainted with flared walls (3619A, makes 31 mm disks) and painted with straight walls (3619, 30 mm, makes 31 mm disks; 3615, 33 mm, makes 35 mm disks; and 3617, 38 mm, makes 40 mm disks). The painted, straight-walled Spec-Caps® can be used in two ways. The simpler is to fill the Spec-Cap® with sample, level it off, and assemble the die around the filled Spec-Caps®

Spec-Cap®. This approach allows many samples to be set up and marked in advance, and when successful is quite efficient, but there are handicaps; perhaps the greatest is that unless the sample material is unusually dense or incompressible, the sample disk will be very thin, prone to cracking and possibly (in the case of low-Z elements) less than "infinitely thick." There is also a chance of the Spec-Cap wall crumpling inward, and the technique of assembling the die around a loaded Spec-Cap requires some finesse.

Alternatively, the flared Spec-Cap technique may be used with either the painted Spec-Caps or the 3619A Pre-Flared Spec-Caps. Here the Spec-Cap is flared before being placed in the die; 3619 and 3617 Spec-Caps are flared by the user with the 3618 Edge-Flaring Tool, 3615 Spec-Caps are flared by the user with the 3625 Edge-Flaring Tool, and the 3619A Spec-Cap is flared at the factory. With this technique the die bore and base are assembled, the lower polished pellet inserted, and the flared Spec-Cap pushed down against that lower pellet.

Then the sample is poured into the die, with the upper polished pellet and the plunger following. During pressing, the sample powder is forced inside the Spec-Cap. When the proper amount of sample is added, the top of the sample disk and the edge of the Spec-Cap will coincide. Obviously the sample weight will vary with the density of the sample and the size of the disk, but the general range of sample weight is, for a 31 mm disk, 5 to 8 grams; for a 35 mm sample disk, 7 to 10 grams; and, for a 40 mm disk, 8 to 12 grams.

BINDERS

Many analytical samples cohere well under pressure; those samples which crumble or ablate after pressing require a binder. Binders are usually blended with the sample after pulverizing and before pressing, but can also serve as a pellet matrix, supporting a thin layer of sample.

Binders can be liquids or powders, and range from commonly available reagents to brand name products with secret formulas. Their use should lead to a stable, crumble-proof sample disk achieved with a minimum of dilution, contamination, and effort.

Generic binders include cellulose, paraffin, boric acid, and graphite. Of these, cellulose and paraffin are available through SPEX SamplePrep in extra-pure, finely powdered form, ideal for blending and pelletizing. Prep-Aid® Cellulose (3642) has a particle size of less than 20 microns, and blends quickly and completely with samples at 10% by weight. It can also be used undiluted as a sample matrix

with the sleeve-and-plunger technique. It will neither stick to a die nor contaminate a sample, and does not cake in the bottle during storage. Disks stabilized with cellulose should, however, be kept in a desiccator if they are to be retained, as with time they can absorb moisture and slowly swell and crack. 25% may be necessary for UltraBind®, an exclusive SPEX SamplePrep product, satisfies our notions of the perfect all-around binder: it blends well with samples, and is self-lubricating, strong when pelletized, and moisture-resistant. Most batches, however, have low amounts of NaCl as a relic of the manufacturing process. A fine (20 µm) powder, UltraBind® blends easily with samples to yield a disk which is easy to remove from the die, and durable enough to withstand rough handling. It also resists cracking and swelling in storage, and is an excellent disk matrix when used with the Sleeve-and-Plunger technique, described in this chapter. UltraBind® (3644) is described on page 143 along with other Prep-Aid binders.

Sample disks formed with 10% to 20% paraffin are air-stable, as the waxy binder seals the surface against moisture. Powdered Paraffin (3646) is also offered by SPEX SamplePrep. As with cellulose, the fine (30 µm) powder blends evenly with samples and does not contaminate them. However, paraffin should not be used undiluted with the sleeve-and-plunger technique, as in this concentration it will stick to the die.

Boric acid is commonly available in pure form, and can be used diluted or undiluted to make a stable sample disk. However, it is much more hygroscopic than cellulose and hence is typically supplied in granular form, requiring grinding in a mill or mortar- and pestle to become a fine enough powder to blend evenly with a sample. Some analysts add boric acid to the sample during the final stage of grinding to achieve this. Sample disks bonded with boric acid must be kept in a desiccator if they are to be preserved.

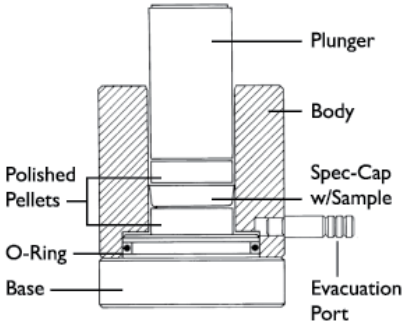
Graphite powder makes sample disks unaffected by moisture, and blends rapidly with any sample. It also serves as its own lubricant, making it easy to free disks from the pellet die. Its disadvantage for XRF is that it must be used in a proportion of at least 50% of the sample disk by weight. Sample disks pressed with graphite are also used for spark ablation techniques.

Binders in general, are designed to cake under pressure or at least flow under pressure and then cake when the pressure is relieved. Hence there is a good chance that adding a binder to the sample before grinding will lead to caking in the grinding container. In this connection it should be noted that a very finely ground sample (e.g.

cement-mix ground to below 10 microns) is more likely to bind well under pressure than the same sample ground to moderate fineness, and that grinding aids are often needed to attain very fine particle size. When a grinding aid such as Vertrel® XF (3650) is used, the binder can usually be added to the sample before grinding.

SELECTING A PELLET DIE

The choice of die is generally determined by the requirements of the analytical instrument. Each SPEX SamplePrep die set is a complete unit. Made of hardened stainless steel for durability and extra wear, the SPEX SamplePrep die includes a body with detachable base, a plunger, and two polished steel pellets. Sample material is pressed in the die bore between the polished pellets, yielding a compact sample disk ready for the spectrometer's sample holder. A convenient "knock-out ring" allows easy extraction of the steel pellets and sample disk from the die. Each precision-machined SPEX SamplePrep die set also incorporates a vacuum hose attachment. This allows evacuation of gases, volatiles, and moisture during pressing, assisting compaction and preventing possible sample disk rupture under vacuum-path conditions. Full instructions for the use of SPEX SamplePrep pellet dies are supplied with each die.



Cross Section of SPEX SamplePrep Die Set with Spec-Cap.

KATANAX FUSION FLUXERS

Our range of Katanax automated electric fusion fluxers are designed to prepare glass disks (beads) for XRF or solutions for ICP/AA. They combine exceptional fusion accuracy with all the advantages of electric power. Katanax fluxers allow you to save time while obtaining the best possible analytical data from fused samples. Our X-Fluxers are robust and versatile to adapt to the needs of today's modern laboratories.

The multi-lingual touch screen interface comes pre-loaded with various fusion programs that can be used as is, or customized for your particular protocol. The interface also allows the user to control and monitor the temperature of the furnace.

Training packages are available for Katanax Fusion Fluxers. Please contact us for further information.

APPLICATIONS

Prepares Lithium borate glass disks for XRF or solutions for AA, ICP, and wet chemistry analysis

Peroxide or pyrosulfate fusions

Liquid or solid oxidations

SAMPLE TYPES

Ceramics
Cement
Minerals
Rocks
Slag
Soil
Clinker
Ores
Catalysts
Refractories
Metals
Alloys

KATANAX X-300 FLUXER

The Katanax X-300 Fluxer® is an electric fusion fluxer, offering new enhanced features for unparalleled results. It is used to prepare glass disks (beads) for XRF analysis and solutions for ICP/AA analysis or for preparing peroxide and pyrosulfate fusions. The X-300 is available as a one, two or three position system. The one and two position systems can be upgraded to the three position system. The three position unit allows you to run up to 3 samples at the same time.

Extraction chimneys allow for direct ventilation of halogens from each crucible position.

Integrated auto-locking safety shield protects the user during the fusion process.

High Performance Furnace with heating elements impervious to flux

Low noise level during heating, melting and standby.

Easy-clean, ceramic mold holders. Mold holder system is user-configurable for 30, 32, 35 or 40 mm molds.

Control panel that adjusts to the user's height and features a USB connection for firmware updates.

SPECIFICATIONS

VOLTAGE	195-250 VAC (50-60 Hz)
DIMENSIONS	19 in. (48 cm) x 20 in. (51 cm) x 25 in. (63 cm)
MAXIMUM POWER	3,000 W
WEIGHT (LBS)	99 lbs. (45 kg)
OPERATING TEMPERATURE	20–1,200 °C
FREQUENCY	50–60 Hz
BREAKER	15 amps

FUSION OPTIONS

Single Position
(X-300M)

Dual Position
(X-300D)

Triple Position
(X-300T)

Molds can be replaced with beakers for solutions.

KATANAX X-600 FLUXER

Built with the most demanding lab in mind, the Katanax X-600 Fluxer® is an electric fusion fluxer offering new enhanced features for unparalleled results. It is used to prepare glass disks (beads) for XRF analysis and solutions for ICP/AA analysis or for preparing peroxide and pyrosulfate fusions. This unit allows you to run up to 6 samples at once.

Easy-clean, ceramic mold holders. Mold holder system is user-configurable for 30, 32, 35 or 40 mm molds.

Integrated auto-locking safety shield protects the user during the fusion process.

Extraction chimneys allow for direct ventilation of halogens from each crucible position.

Low noise level during heating, melting and standby.

High Performance Furnace with heating elements impervious to flux.

Control panel that adjusts to the user's height and features a USB connection for firmware updates.

SPECIFICATIONS

VOLTAGE	195-250 VAC (50-60 Hz)
DIMENSIONS	56 cm (22 in.) x 105 cm (41 in.) x 69 cm (27 in.)
MAXIMUM POWER	4,000 W
WEIGHT (LBS)	187 lbs. (85 kg)
OPERATING TEMPERATURE	20–1,200 °C
FREQUENCY	50–60 Hz
BREAKER	Built in 20 amps

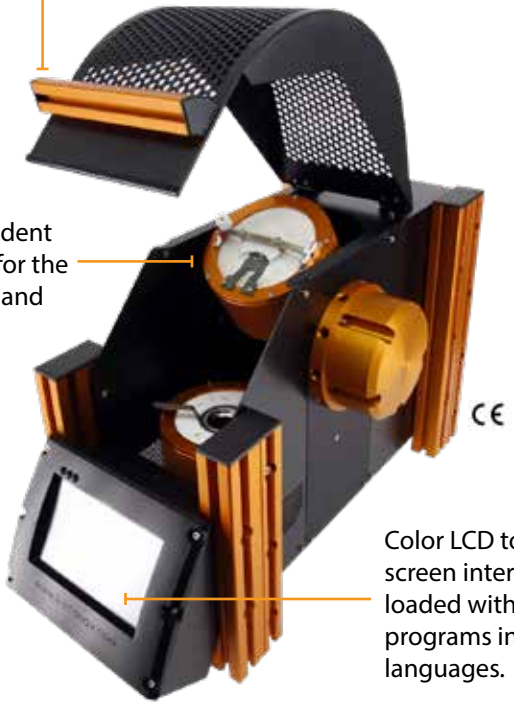
FUSION OPTIONS

Optional solutions stirrer with variable speed allows the X-600 to produce solutions in up to 6 beakers simultaneously.

KATANAX K-1 PRIME

The K1 Prime is a single-position, fully automated, electric fusion machine. It is ideal for preparing fused beads for XRF analysis and solutions for AA, ICP, and wet chemistry analysis. Automated control of fusion parameters including heating time and temperature, mixing time and rate, cooling time for glass disks or stirring time for solutions. Model numbers K1P-30, K1P-32, K1P-35 and K1P-40 with 30 mm, 32 mm, 35 mm and 40 mm mold holders, respectively.

Safety shield protects users from the heat.



Independent heaters for the crucible and mold.

Color LCD touch-screen interface pre-loaded with fusion programs in multiple languages.

SPECIFICATIONS

VOLTAGE	110-127 VAC 220-240 VAC (50-60 Hz)
DIMENSIONS	19 in. (47 cm) x 11 in. (28 cm) x 26 in. (66 cm)
WEIGHT (LBS)	66 lbs. (30 kg)
OPERATING TEMPERATURE	20–1,150 °C
POWER CONSUMPTION	1,300 W
POWER CORD	3-prong grounded cord, 115 V 60 Hz or 2-prong French/German style plug with an IEC connector allowing multi-country power cables.

FUSION OPTIONS

- Fused Beads
- Solutions

ACCESSORIES

CRUCIBLES & MOLDS FOR KATANAX® FLUXERS

SPEX SamplePrep offers crucibles made of 95% platinum and 5% gold, the standard non-wetting alloy for borate fusions. Zirconium crucibles are available for peroxide fusions. Pt/Au crucibles and molds are offered in regular and heavy-duty versions; heavy-duty platinumware lasts longer and is less likely to warp. All platinumware sold separately from the instrument. Price quotations are available upon request by contacting Customer Service. Please note that prices are quoted daily and may change due to market fluctuations.

POLISHING KIT

This polishing kit is used to restore the shiny finish to platinumware of any shape and size. It includes an electric hand held rotary tool with flexible shaft, a table stand, buffing pads and diamond pastes of assorted grades.

Before After

This kit is available in :

115 VAC / 60 HZ VERSION
(p/n KP9004A)

230 VAC / 50 HZ VERSION
(p/n KP9005A)

230 VAC / 60 HZ VERSION
(p/n KP9006A)

CRUCIBLES

KP1001A REGULAR CRUCIBLE, 30 ML CAPACITY
95% Pt/5% Au, 26 g. Straight-walled crucibles.

KP1002A HEAVY-DUTY CRUCIBLE, 30 ML CAPACITY
95% Pt/5% Au, 30 g. Straight-walled crucibles with a thicker rim and bottom for greater durability.

KP0049A ZIRCONIUM CRUCIBLE, 30 ML CAPACITY
100% Zirconium, 55 g. Straight-walled crucible.

KP1101A FLARED RIM CRUCIBLE, 30 ML CAPACITY
95%Pt/5% Au, 28 g. Regular weight.

KP1112A FLARES RIM CRUCIBLE, 30 ML CAPACITY
95%Pt/5% Au, 40 g. Heavy weight.

MOLDS (nominal weights)

KP1003A MOLD – 30 MM
95% Pt/5% Au, 18-21 g.

KP1004A HEAVY-DUTY MOLD – 30 MM
95% Pt/5% Au, 27-30 g.

KP1005A MOLD – 32 MM
95% Pt/5% Au, 21-23 g.

KP1006A HEAVY-DUTY MOLD – 32 MM
95% Pt/5% Au, 35-37 g.

KP1007A MOLD – 35 MM
95% Pt/5% Au, 25-26 g.

KP1008A HEAVY-DUTY MOLD – 35 MM
95% Pt/5% Au, 39-41 g.

KP1009A MOLD – 40 MM
95% Pt/5% Au, 34-35 g.

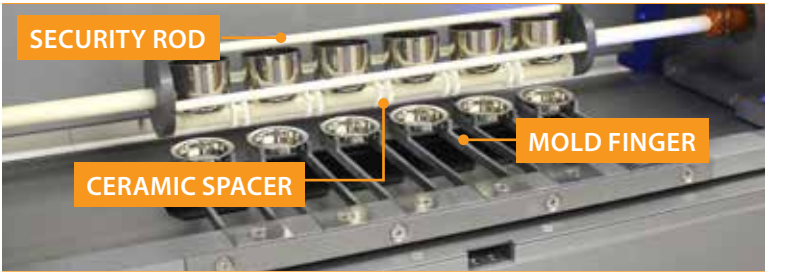
KP1010A HEAVY-DUTY MOLD – 40 MM
95% Pt/5% Au, 47-48 g.

X-600 ACCESSORIES

BEAKERS

KP7010A - TEFLON® BEAKER
Square PTFE (Teflon) beaker with magnetic bar, X-600

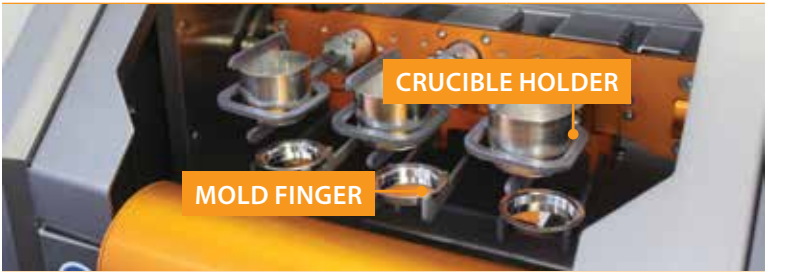
KP7010S - TEFLON® BEAKER
Square PTFE (Teflon) beakers with magnetic bar (set of six), X-600



KP7500A Spare kit basic

- 10 ceramic washers
- 5 ceramic spacers (5.99 mm)
- 6 ceramic spacers (41 mm)
- 4 ceramic tubes (312.5 mm)
- 4 security rods1 thermocouple
- 2 heating elements
- 3 mold finger pairs
- 2 ceramic axles

X-300 ACCESSORIES



KP8500A Spare kit basic

- 1 Heating element
- 1 Crucible holder (hoop + fork)
- 1 Mold finger pair
- 1 Thermocouple

KP0010A BEAKER PTFE
(Teflon) with magnetic bar, X-300

HANDLING TOOLS FOR USE WITH MUFFLE FURNACES

7151T LONG TONGS
For handling crucibles; 20 in.

7154 SHORT TONGS
For handling crucibles; 9 in.

7151R RACK AND TONGS
For holding six 7152 or 7152HP graphite crucibles.Ni-Cr (nichrome) alloys used for rack withstand temperatures up to 1200 °C.

GRAPHITE CRUCIBLES FOR USE WITH MUFFLE FURNACES

7152 GRAPHITE CRUCIBLES
31.8 mm O.D. x 28.6 mm, 8.4 mL capacity. Regular purity graphite; sold in packs of 100.

7152HP GRAPHITE CRUCIBLES
31.8 mm O.D. x 28.6 mm, 8.4 mL capacity. High purity graphite; sold in packs of 10.

7155 GRAPHITE CRUCIBLES
High-purity graphite. 38.1 mm O.D. x 46.0 mm tall. 25.4 mm I.D. with flat bottom. 20 mL capacity. Sold in packs of 10.

7156 GRAPHITE CRUCIBLES
Regular-purity graphite crucible. 38.1 mm O.D. x 46.0 mm tall.25.4 mm I.D. with flat bottom. 20 mL capacity. Sold in packs of 10

7157 GRAPHITE CRUCIBLES
High-purity graphite crucible also capable of casting 31 mm glass disks. 44.4 mm O.D. x 38.1 mm tall. 31 mm I.D. with flat bottom. 27 mL capacity. Sold in packs of 10.

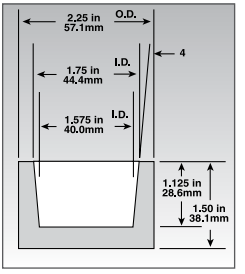
7158 GRAPHITE CRUCIBLES
Regular-purity graphite crucible also capable of casting 31 mm glass disks. 44.4 mm O.D. x 38.1 mm tall. 31 mm I.D. with flat bottom. 27 mL capacity. Sold in packs of 10

7159 GRAPHITE CRUCIBLES

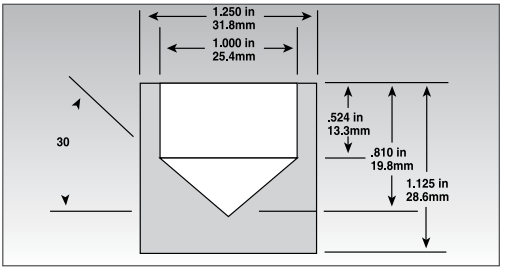
High-purity graphite crucible also capable of casting 33.7 mm glass disks. 50.8 mm O.D. x 38.1 mm tall. 33.7 mm I.D. with flat bottom. 32 mL capacity. Sold in packs of 10

7161 GRAPHITE CRUCIBLES

For casting 40 mm glass disks, 57.1 mm O.D. x 38.1 mm, 44.5 mL capacity, 40 mm I.D. flat bottom. High purity graphite; sold in units of 10.



7161 Graphite Crucible



7152, 7152 HP Graphite Crucible

Please note the crucibles are not compatible with Katanax Fusion Fluxers.

FUSION FLUX

SPEX SamplePrep offers a full line of Fusion Fluxes and additives. These fluxes have exceptional qualities due to their purity and coarse glass-bead texture.

Some of these fluxes include lithium bromide or lithium iodide as a non-wetting agent. A small amount of LiBr or LiI is added to the flux when making a "glass bead" (lithium borate sample disk), to keep the melt from sticking to the crucible or mold. Additional LiBr or LiI is added to the flux before making a solution for ICP/AA analysis, so the entire melt is transferred to the beaker. LiBr solution in a dropper bottle is a convenient way to add non-wetting agent to a fusion, at 6-7 mg per drop. Please call SPEX SamplePrep for a free sample of flux, or information on a custom flux.

MIXTURE	COMPOSITION (%)	PURE	ULTRA-PURE
LiT	100.00	FFB-1000-02	FFB-1000-03
LiT/LiBr	99.50/0.50	FFB-1005-02	FFB-1005-03
LiT/LiI	99.50/0.50	FFB-1007-02	FFB-1007-03
LiT/LiM	67.00/33.00	FFB-6700-02	FFB-6700-03
LiT/LiM/LiBr	66.67/32.83/0.50	FFB-6705-02	FFB-6705-03
LiT/LiM/LiI	66.67/32.83/0.50	FFB-6707-02	FFB-6707-03
LiT/LiM	50.00/50.00	FFB-5000-02	FFB-5000-03
LiT/LiM/LiBr	49.75/49.75/0.50	FFB-5005-02	FFB-5005-03
LiT/LiM/LiI	49.75/49.75/0.50	FFB-5007-02	FFB-5007-03
LiT/LiM	35.00/65.00 (12/22)	FFB-3500-02	FFB-3500-03
LiT/LiM/LiBr	(11.94/21.89/0.17)	FFB-3505-02	FFB-3505-03
LiM	100.00	FFB-0000-02	FFB-0000-03
LiM/LiBr	99.50/0.50	FFB-0005-02	FFB-0005-03
LiM/LiBr	98.50/1.50	FFB-0007-02	FFB-0007-03

ADDITIVE	PART NUMBER
LiBr crystal (125g)	FFB-100-03
LiBr – 15mL solution	FFB-103-03
LiBr – 15mL solution (10pk)	FFB-105-03
LiI – 15mL solution	FFB-113-03
LiF crystals (125g)	FFB-200-03
$LiNO_3$ (250g)	FFB-300-03
$LiNO_3$ (500g)	FFB-301-03

APPLICATION NOTES

To access our full library and download the complete application note visit www.spexsampleprep.com/appnotes.

SP026: PREPARATION AND XRF ANALYSIS OF FLY ASH BEADS APPLICATION: CEMENT, SLAG, & FLY ASH

PREPARATION AND XRF ANALYSIS OF FLY ASH FUSED BEADS ABSTRACT

Fly ash is a waste product from the combustion of coal and is comprised of the mineral particles that rise with the flue gases. Recovered fly ash is used as a component in certain cement mixes and improves the durability and strength of concrete.

In this study, a sample of fly ash was pulverized and blended, and then fused into two sets of glass beads using a Katanax single position electric fusion fluxer. Two different lithium borate fusion fluxes were used to prepare five beads with each flux. The beads were then analyzed by energy dispersive X-ray florescence spectroscopy (EDXRF) and the results were compared both within a set and between the two sets to evaulate consistency of beads produced using the fluxer and effect of flux composition, on XRF measurements.

HT004: SAMPLE PREPARATION OF CEMENT SAMPLES BY FUSION

ASTM C-114 ACCREDITATION FOR CEMENT ANALYSIS BY FUSION

Cement industries, as well as other types of factories that make products made of raw materials, need to meet more stringent quality controls. The physical properties of the final products can be influenced if the composition changes for any reason in the fabrication process. To ensure maximum quality of the final product, standards are available that help to ensure minimal variations in the analytical process.

This standard is dedicated to the analysis of hydraulic cement. It gives limit values to conform to, ensuring that the analytical process is fully controlled and yields minimal variations in results. It suggests reference test methods for every element of interest in the analysis of cement. But one can also use a "rapid test method" if the variation in results conform to the limitation proposed by this norm. The "rapid test method" can be any method used to determine the concentration of analytes that complies with the ASTM C-114 validation requirements. This means that one can use fusion as a rapid test method if the resulting variations are below the allowed values given by ASTM C-114 for the appropriate elements. The method needs to be validated with acceptable certified reference materials (CRMs) provided by National Institute of Standard and Technology (NIST) as suggested in the ASTM C-114 standard.

BORATE FUSION

Borate fusion is an extremely effective method of preparing cement, refractories, ceramics, rock, and similar materials for elemental analysis by XRF, AA, and ICP. The samples are mixed in powdered form with a flux, either lithium tetraborate or a mixture of lithium tetraborate and lithium metaborate. The sample mixture is heated until the flux melts and the sample dissolves in it, yielding a homogenous melt. The sample forms borate salts and this eliminates particle size and mineralogical effects. The use of borate fusions will also minimize matrix effects which are commonly seen in XRF analysis. The melt can be cast as a glass disk for XRF, or quickly dissolved in dilute nitric acid or hydrochloric acid for analysis in solution by AA or ICP. Recent advances also allow borate fusion of samples containing sulfides, ferroalloys, etc.

One of the advantages of borate fusion is the short preparation time, typically ten to fifteen minutes to make glass disks or solutions with automated fluxers. For samples prepared as liquids, borate fusion can be quicker overall than microwave dissolution in pressure vessels, and the use of hazardous acids (e.g. HF) avoided.

SPEX SamplePrep offers two approaches to borate fusion. The Katanax X-Fluxer Series Automated Electric Fluxers for rapid, reproducible fusions, and graphite crucibles for smaller-scale operations with muffle furnaces. SPEX SamplePrep also provides the full line of SPEX Fusion Flux.

WHEN TO EMPLOY BORATE FUSION

Borate fusions are widely used for samples which are either difficult to prepare as homogenous pressed powders (e.g. cement), hard to dissolve in acid (e.g. zirconia and alumina), or both (e.g. metal ores and silicate rocks). For borate fusions to be successful, the sample when fused must be in the form of an oxidized, inorganic compound. Cement is usually a blend of carbonates and silicates; zirconia and alumina are oxides; and so forth. Compounds without oxygen such as sulfides, carbides, chlorides, etc, must be oxidized before being fused. Reduced metals must also be oxidized. Organic compounds must be ashed. (An example of this is the analysis of metallurgical coal. The coal sample must be ashed and the ash is

then fused. The sulfur in the coal will volatilize in the process and therefore sulfur cannot be measured). Once fusion is complete, the melt can be cast as a glass disk or poured into a dilute acid solution and dissolved. Platinum-group metals cannot be fused with borates because these compounds reduce during fusion and the metals will not only remain insoluble in the flux but can also alloy with the 95% platinum/5% gold crucibles and damage or destroy them.

Many users of the X-300 and X-600 X-Fluxer Series Automated Electric Fluxers are in the following industries: cement, glass, ceramics, mining and minerals. Samples analyzed include not only raw materials like dolomite, sand, basalt and iron ore, but also their industrial products and by-products such as cement, building materials and mining concentrates. Additional industrial samples include pigments such as TiO2, and slags from smelters, blast furnaces, refineries and glass plants. Most of these samples are naturally oxygen-rich and do not require chemical transformation prior to borate fusions. However, hybrid oxidation/fusion techniques have been developed for reliable borate fusions of sulfides, carbides and some ferro-alloy materials formerly considered out-of-bounds for the technique.

Borate fusion has become increasingly popular as a preparation technique for XRF sample disks. Because fusion eliminates particle size and mineralogical effects and produces a homogeneous sample, it has proven to be the best method for materials that have these characteristics when X-ray fluorescence analysis is the method of choice. The fusion method will reduce matrix effects but not eliminate them. Borate fusion may not be the method of choice if the analyst is interested in trace analysis, since the sample is diluted during fusion. There have been recent advances in improving the performance of fusions for trace analysis.

The pressed powder method can be highly accurate when carefully done with multi-phase samples such as cements, rock, sand and ore but such samples are subject to segregation during grinding and pressing, and to matrix effects (e.g. Particle size and mineralogical effects). Borate glass disks are easier to preserve than pressed powder disks because they are stable if carefully stored in desiccators. Synthetic standards for XRF can also be made from pure oxides with the borate fusion method, as borate glass is essentially a solid solution with few matrix-matching problems.

In preparing samples for AA, ICP, and other liquid-analyzing techniques, the major advantage of borate fusion is that it is often simpler and quicker than dissolution with acid in a microwave

pressure vessel. A complete fusion/solution procedure, from ignition of the heating elements to decanting of a clear solution, can take fifteen minutes or less in an automated fluxer. While borate fusions do require some caution in evacuating heat and fumes, and the use of dilute HCl or HNO_3 to dissolve the melt, hazardous reagents such as HF and other concentrated mineral acids are not necessary.

Borate fusion methods offer a wide range of applications but may not be suitable for all materials. Fusion destroys the original form of the sample, so structural and molecular information should be measured before the fusion is made. The high temperature of borate fusion (1000 to 1150 ˚C) drives off compounds of volatile metals such as Hg, Sn, and Sb, while other compounds form during fusion. Extra steps necessary to prepare organic materials and reduced inorganics for fusion can extend turnaround time but still may be the most accurate method to choose. For many samples borate fusion is the simplest, quickest, and most accurate analytical approach.

Both fusion and pulverizing/pressing are important and widely used sample preparation methods, each with their own advantages. SPEX SamplePrep has a full range of equipment for either approach. Please consult our applications specialists to help determine which method is more suitable for your laboratory.

HOW TO PREPARE A FUSED SAMPLE

Fusions are accomplished in several steps. First the sample is mixed with a flux in an appropriate ratio (usually between 1:2 and 1:10), with the addition of a non-wetting agent to prevent the flux from sticking to the crucible and the mold. Typical amounts of flux/sample mixture are 6 to 7 grams for a 31 mm glass disk, and 1 to 2 grams for a solution. The sample is heated past the melting point of the flux in an inert, heat-resistant crucible. Most borate fusions are performed in crucibles made of 95% platinum and 5% gold, a standard non-wetting alloy. Some borate fusions are done in graphite crucibles.

During the fusion, heating is maintained and the crucible regularly agitated until the sample has completely dissolved in the molten flux. At this point the melt is either poured into a mold and annealed to form a glass disk for XRF, or poured into dilute mineral acid (e.g. 10% HNO3) and stirred until the glass flux dissolves. In some cases (notably pyrosulfate fusions) the melt is left to harden in the crucible, and the crucible and the glass together are placed in an acid solution to dissolve the glass.

It is possible to achieve a high sample throughput with an automated, programmable fluxer. The Katanax X-Fluxers, when

programmed for cement samples, can produce up to several fused glass disks in an hour, using 0.6 grams of cement mixed with 6 grams of flux and about half a percent of LiBr, a non-wetting agent.

The same borate fusion procedures can be carried out manually in a muffle furnace with SPEX SamplePrep graphite crucibles. The larger flat-bottomed SPEX SamplePrep crucibles can be used to cast glass disks as well as perform fusions, while the crucibles are handled and agitated with SPEX SamplePrep tongs. The time per sample with the muffle furnaces is usually much longer than with an automated fluxer. Each approach has its advantages. Please contact our applications specialists to determine the optimum equipment for your requirements.

HOW TO SELECT A FUSION FLUX

Most fusions involve the use of lithium tetraborate ($Li_2B_4O_7$, M.P. 920 ˚C), lithium metaborate ($LiBO_4$, M.P. 845 ˚C), or a mixture of the two. As a rule lithium tetraborate is better suited for the dissolution of basic oxides, and is preferred for cement and most ores. Lithium metaborate or "met/tet" mixtures are more suitable for acidic oxides such as silicate rocks and silica-alumina refractories. Individually or together, these lithium borates will dissolve oxides, carbonates, silicates, sulfates, etc. Metals, sulfides, nitrates, carbides, phosphides, etc. cannot be fused directly in lithium borate fluxes, and will often attack platinum-gold crucibles, or alloy with them. However, many of these materials can be first oxidized with standard techniques and then successfully fused. Methods have been developed for fusing sulfide-rich material such as copper ore. The sample is mixed with lithium or sodium nitrate and preheated to oxidize the sulfide to sulfate. When this has been done the fusion can proceed as normal without any loss of sulfur from the fusion.

Other fluxes include sodium tetraborate ($Na_2B_4O_7$), sodium metaphosphate ($NaPO_3$), and potassium pyrosulfate ($K_2S_2O_7$). These have lower melting points than lithium borate fluxes and more specialized applications. Melting point may be a factor in the selection of a flux, as the higher temperature of a fusion, the greater the degree of volatilization. However the utility of lithium tetraborate and lithium tetraborate/metaborate mixtures is so great that most analytical fusions are carried out with these fluxes at temperatures between 1000 and 1150 ˚C.

For further reading please refer to:
Physics and Chemistry of Borate Fusion, Theory and Application
available at www.spexsampleprep.com/boratefusionbook
or request a copy from learnmore@spex.com.

ADDITIVES FOR FUSIONS: NON-WETTING AGENTS, FLUIDIZERS, AND OXIDIZERS

Non-wetting agents (NWA) are iodides and bromides which can be added in small quantities to a fusion so the molten flux will not stick to the crucible or mold. The non-wetting agent increases the surface tension of the melt. A fused disk with too little NWA will have a concave upper surface and may be difficult to remove from the mold, whereas a molten flux bead with excessive NWA will ball up when poured and not form a complete disk.

When glass disks for XRF are being made, NWA is mixed with the flux and the sample before fusion starts. Typically the amount of NWA is about 0.2% of the weight of the flux, e.g. 12 mg of NWA for 6 grams of flux. Certain samples such as iron ores, which greatly increase the "stickiness" of a melt, require additional NWA. As non-wetting agents gradually volatize during a fusion, longer fusions may also need greater amounts of NWA. The ideal amount of NWA for a specific procedure is usually determined by experiment.

When making solutions by pouring the molten flux into a dilute mineral acid, it is desired to have complete transfer from the crucible to the beaker. This can require a much higher proportion of nonwetting agent than is necessary to pour a glass disk. A quantity of flux plus sample not exceeding 2 grams might require 50 to 100 mg of NWA. Lithium iodide and bromide are popular non-wetting agents because they do not add an impurity to the flux. However lithium bromide is hygroscopic, so it is usually made into a saturated solution and added to the flux from a dropper bottle. Lithium iodide and sodium iodide, are more air-stable, and easier to use as solids. While it is simpler to add a drop or two of NWA than it is to weigh out 10 or 20 mg of a solid, liquid NWA cannot be added to a hot crucible while a fusion is in progress. Lithium fluoride can be used as a fluidizer, lowering the melting point of a flux and making it flow far more easily. At 10% by weight, it lowers a flux's melting point by about 100 ˚C.

Oxidizers such as lithium nitrate and sodium nitrate are useful in eliminating unoxidized components from a sample that will not fuse. Graphite, often present in cement mix, is relatively harmless but can leave a black film on a glass disk. Graphite can be oxidized to CO2. Other sample components such as phosphides and sulfides may be corrosive enough to damage a crucible in a single fusion. If they are oxidized to phosphates and sulfates they will be comparatively harmless, and their cations will be present in the fused glass disk for analysis. As oxidizers have much lower melting points than borate fluxes, any fusion including them should proceed at a low temperature until oxidation is complete.

ACCESSORIES

The XRF spectroscopist is often confronted with a variety of samples. Liquid samples, from water to oil, require virtually no preparation. Many solid samples, if homogeneous, can be machined to the proper shape and run directly. Others, such as refractories, ores, and biological materials, are too inhomogeneous, and must be pulverized or fused. Then the homogenized material can be dissolved for analysis as a liquid sample, pressed into pellet form, or cast as a glass-like solid.

SPEX SamplePrep provides disposable sample-handling accessories for powder and liquid XRF samples. Powder samples for XRF can be pressed and protected in the SPEX SamplePrep Spec-Cap®, an aluminum cap for reinforcing pellets. Liquid XRF samples can be run in our X-CELLS®. These XRF products listed save you time and money, assuring reproducible, contamination-free results.

SPEC-CAPS®

SPEX SamplePrep® Spec-Caps® are shallow, thin-walled aluminum cups which are routinely used in the production of pressed powder sample disks for OES, XRF, and other analytical techniques. The Spec-Cap forms the bottom and sides of the finished pellet. Thus reinforced, sample disks are resistant to chipping and breaking, and are more easily handled, marked, and stored than unclad disks.

3615 SPEC-CAP®

33 mm wide x 8 mm deep, for 3616 die. Produces pellets 35 mm x 5 mm. Outside painted to prevent seizure in die and to facilitate labelling. Sold in units of 100 or 1000 (3615M).

3617 SPEC-CAP®

38 mm wide x 9 mm deep, for 3614 die. Produces pellets 40 mm x 5 mm. Outside is painted to prevent seizure in die and to facilitate labelling. Sold in units of 100 or 1000 (3617M).

3619 SPEC-CAP®

30 mm wide x 8 mm deep, for 3623 die. Produces pellets 31 mm x 5 mm. Outside painted to prevent seizure in die and to facilitate labelling. Sold in units of 100 and 1000 (3619M).

3619A PRE-FLARED SPEC-CAP®

31 mm wide x 8 mm deep, for 3623 die. Produces pellets 31 mm x 5 mm. Preflared for a snug fit in the 3623, 31 mm Evacuable Pellet Die, and has an unpainted surface. May be substituted for the 3619, 30 mm Spec-Cap in applications where flaring is recommended. Sold in units of 100 and 1000 (3619AM).

EDGE FLARING TOOLS

3618 EDGE-FLARING TOOL

For 3617 and 3619 Spec-Caps. This tool widens the Spec-Cap rim to ensure a snug fit against the walls of the die, thus preventing sample loss and pellet jamming.

3625 EDGE-FLARING TOOL

For 3615 Spec-caps. This tool widens the Spec-Cap rim to ensure a snug fit against the walls of the die, thus preventing sample loss and pellet jamming.

PREP-AID® BINDERS

3642-150 PREP-AID CELLULOSE BINDER – $[C_6H_{10}O_5]n$

≤ 20 µm powder, 150 g bottle. Can be blended with sample at 10% by weight to form XRF sample disk, or used undiluted as sample matrix with Sleeve-and-Plunger Set. Will bond 200-250 disks or provide matrix for 25-35 supported sample disks (3642-450).

3644-150 PREP-AID ULTRABIND®

≤ 20 µm powder, 150 g bottle. Can be blended with sample at 10% by weight to form XRF sample disks, or used undiluted as sample matrix with Sleeve-and-Plunger Set. Combines moisture resistance of Paraffin Binder with the ease of removing a prepared disk from the die that is for the Cellulose Binder. Bonds 200–250 disks or provide matrix for 25–35 supported-sample disks. Also available as a 450 g bottle (3644-450).

3644T-500 ULTRABIND® TABLETS

UltraBind® is now available as 0.5 g tablets for quick and easy sample preparation. Simply blend with sample (~10% wt/wt) and press into pellet for XRF analysis. Available in jars of 500 (3644T-500) or 1,500 (3644T-1500) tablets.

3646-150 PREP-AID PARAFFIN BINDER – (C_NH_{2N+2})

≤ 30 µm powder, 150 g bottle. Can be blended with sample at 10% by weight to form XRF sample disk, or used undiluted as sample matrix with Sleeve-and-Plunger Set. Will bond 200-250 disks or provide matrix for 25-35 supported sample disks. Also available as 450 g bottle (3646-450).

X-CELLS®

3527 X-CELL®

40 mm Closed X-Ray cell with polyethylene snap-ring and cup with snap-post vent and reservoir. Requires window film. Sold in units of 100

3527I BUBBLE-FREE CELL INSERT

Patented insert for the 3527 40 mm X-Cell®. Prevents trapped air from forming a bubble under the window film when the X-Cell® is used in the upright position. Sold in units of 100.

3529 X-CELL®

31 mm Closed X-Ray cell with polyethylene snap-ring and cup with snap-post vent and reservoir. Requires window film. Sold in units of 100.

3561 X-CELL®

Polyethylene snap-ring, collar, and cup with snap-post vent and reservoir. Requires window film. Sold in units of 100.

3565 X-CELL®
43 mm Closed X-Ray Cell for Horiba Sulfur Analyzers. Polyethylene snap-on collar and cup with vent and reservoir. Requires window film. Sold in units of 100.

3571 X-CELL®
31 mm Double Open End X-Ray Cell with Collar two polyethylene snap-rings, collar, and open body. Requires window film.
Sold in units of 100.

3577 X-CELL® 31 MM
Micro X-ray Cell with Collar. Two polyethylene snap-rings, collar, and open body. Unique cell for small, air-sensitive or hazardous samples. Requires window film. Sold in units of 100.

X-CELL® NUMBER	DESCRIPTION	OVERALL HEIGHT	OVERALL O.D.	WINDOW PLANE O.D.	APERTURE	CAPACITY (ML)
3529	31 mm Closed X-Cell®	22.0	32	30.5	24.5	8.0
3561	31 mm Universal X-Cell®	22.0	31.6	31.6	24.5	8.0
3571	31 mm Open End X-Cell®	22.0	31.6	31.6	24.5	1.0
3527	40 mm X-Cell®	22	39.5	38.0	31.5	13
3565	43 mm X-Cell®	19	48	43.0	38.8	11.5*
3577	Micro X-Cell®	22	31.6	31.6	6.3	0.5

Note: All measurements in mm unless otherwise noted

SPEX SamplePrep X-CELLS® are designed to fit the major brands of XRF spectrometers such as PANalytical, Rigaku, Bruker, and Thermo Instruments. The requirements of the application will dictate the type and size of X-CELL®and which window material to use.

THIN FILM

3511 KAPTON® WINDOW FILM (ROLL)

0.3 mil (8 µm) thick. Supplied as continuous roll 2 7/8 in. (73 mm) wide, 50 ft. (15 m) long, in box with serrated edge for tearing film to desired length. (Polyimide)

3512 KAPTON PRE-CUT CIRCLES

0.3 mil (8 µm) thick. Supplied as pre-cut circles 2 1/2 in. (63.5 mm) diameter, 500 sheets per box.

3515 MYLAR® PRE-CUT CIRCLES (LARGE)

0.25 mil (6 µm) thick. Supplied as pre-cut circles 3 1/4 in. (82.6 mm) diameter, 500 sheets per box.

3518 MYLAR PRE-CUT CIRCLES

0.25 mil (6 µm) thick. Supplied as pre-cut circles 2 1/2 in. (63.5 mm) diameter, 500 sheets per box.

3517 MYLAR WINDOW FILM (ROLL)

0.25 mil (6 µm) thick. Supplied as continuous roll 2 7/8 in. (73 mm) wide, 300 ft. (92 m) long, in box with serrated edge for tearing film to desired length.

3516 MYLAR WINDOW FILM (ROLL)

0.12 mil (3 µm) thick. Supplied as continuous roll 2 7/8 in. (73 mm) wide, 300 ft. (92 m) long, in box with serrated edge for tearing to desired length. (Polyester)

3520 POLYPROPYLENE WINDOW FILM

0.2 mil (5 µm) thick. Supplied as continuous roll 2 7/8 in. (73 mm) wide, 300 ft. (92 m) long.

3521 POLYPROPYLENE PRE-CUT CIRCLES

0.2 mil (5 µm) thick. Supplied as pre-cut circles 2 1/2 in. (63.5 mm) diameter, 500 sheets per box.

3525 ULTRALENE® WINDOW FILM (ROLL)

0.16 mil (4 µm) thick. Supplied as continuous roll 2 7/8 in. (73 mm) wide, 300 ft. (92 m) long, in box with serrated edge for cutting film to desired length.

3526 ULTRALENE® PRE-CUT CIRCLES

0.16 mil (4 µm) thick. Supplied as pre-cut circles 2 1/2 in. (63.5 mm) diameter, 500 per box with paper interleaving.

FILM P/N	DESCRIPTION	THICKNESS (MIL)	THICKNESS (MICRON)	TYPE	QUANTITY	DIAMETER
3511	Kapton	.3	8	Roll	50 ft (15mm)	-
3512	Kapton	.3	8	Precut	500 Circles	2 1/2 in. (63.5mm)
3515	Mylar	.25	6	Precut	500 Circles	3 1/4 in. (82.6mm)
3516	Mylar	.12	3	Roll	300 ft (92m)	-
3517	Mylar	.25	6	Roll	300 ft (92m)	-
3518	Mylar	.25	6	Precut	500 Circles	2 1/2 in. (63.5mm)
3520	Polypropylene	.20	5	Roll	300 ft (92m)	-
3521	Polypropylene	.20	5	Precut	500 Circles	2 1/2 in. (63.5mm)
3525	Ultralene	.15	4	Roll	300 ft (92m)	-
3526	Ultralene	.15	4	Precut	500 Circles	2 1/2 in. (63.5mm)

XRF ANALYSIS TECHNICAL INFORMATION

INTRODUCTION TO XRF ACCESSORIES

The XRF spectroscopist is confronted with an almost infinite variety of samples. What they have in common is that they must all be presented to the XRF spectrometer in the form of a homogeneous, flat disk. Liquid samples, from water to oil, are run in sample cells covered with a thin film. Many solid samples, if homogeneous, can be machined to the proper shape and run directly. Others, such as refractories, ores, and biological materials, are too inhomogeneous and must be pulverized or fused. Then the homogenized material can be dissolved for analysis as a liquid sample, pressed into pellet form, or cast as a glass-like solid.

SPEX SamplePrep manufactures disposable sample-handling accessories for powder and liquid XRF samples. Powder samples for XRF can be pressed and protected in the SPEX SamplePrep Spec-Cap®, an aluminum cap for reinforcing pellets. Liquid XRF samples can be run in SPEX SamplePrep X-CELLS®. These and the related products listed here will save you time and money, and assure you of reproducible, contamination-free results.

SPEX SamplePrep also provides an extensive line of sample preparation equipment particularly suited for XRF spectroscopy. There are laboratory mills and grinding containers capable of converting most solid samples into homogeneous powders, as well as dies and presses capable of compressing those powders into flat, uniform sample disks, ready for XRF analysis. There are also the Katanax X-Fluxer Automated Electric Fluxers for rapid, automated borate fusions, yielding XRF glass disks, or solutions for AA, ICP, etc. For further fusion work there are also full lines of graphite crucibles, borate fusion fluxes, and fusion additives such as non-wetting agents and oxidants. These products are described elsewhere in this handbook; please feel free to consult our sample preparation specialists to determine the optimum equipment and techniques for your application.

REINFORCING XRF PELLETS WITH SPEC-CAPS®

SPEX SamplePrep Spec-Caps® are shallow, thin-walled aluminum cups which are routinely used in the production of pressed-powder sample disks for XRF. The Spec-Cap® forms the bottom and partial sides of the completed sample disk (also called pellet, briquette, or planchet). Thus reinforced, the disks are resistant to chipping

and breaking and are more easily handled, marked and stored than unclad disks.The sample holders of most X-ray spectrometers will accept 31 mm (11/4 in.) sample disks. For these instruments, 3619 and 3619A Spec-Caps® are ideal. Several spectrometer manufacturers have shifted toward 35 mm and 40 mm sample disks, and for these larger sizes we recommend 3615 and 3617 Spec-Caps®, respectively.

X-CELLS® FOR XRF LIQUID SAMPLES

SPEX SamplePrep X-CELLS® are used throughout the world for fast, dependable, reproducible running of liquid samples in XRF spectrometers. They are designed to fit into the major suppliers of XRF spectrometers such as PANalytical, Rigaku, Bruker, and Thermo Instruments. The requirements of the application will dictate the type of X-CELL® and which window material to use. Made of contaminant-free polyethylene, closed X-CELLS® are available in both 31 mm and 40 mm sizes to fit most spectrometers. For special applications there are 31 mm Double Open End and Micro X-CELLS®, and a 43 mm Closed Cell for Horiba Sulfur Analyzers. The patented closed X-CELL®design features a snap-open seal for venting volatile samples and a reservoir cup for catching any liquids that expand through the vent. This assures the analyst of a smooth, flat window for improved precision, yet guards against spillage in inverted-optic spectrometers. A roughened surface within the reservoir cup facilitates labeling. Each closed X-CELL®consists of a cup and snap-on ring. For routine liquid samples, the cup is filled with liquid, a piece of film is spread over the top, then the snap-ring is pushed down over the cup to form a flat drum-like window. The cell is turned over and, if desired, the snap-post on the rear can be pushed to break the seal and to permit venting of trapped bubbles or expanded liquid while the sample is run. The filled X-CELL® is then positioned in the spectrometer mask.

X-CELLS® can also be used to run solution residues and small quantities of pastes or powders. For solution residues, the film window is prepared as for solutions, then dished slightly with the rounded end of a glass rod. A drop of solution placed there and warmed under an IR lamp dries to a smudge for in situ analysis. If powder or paste is spread on the surface and another piece of film overlaid before snapping on the ring, the sandwiched sample is ready for X-ray analysis. The 3527 40 mm X-CELL®can also be used to hold crumbling 31 mm sample disks.

SPEX SamplePrep also supplies versatile Double Open End X-CELLS® which are ideal for many unusual or hard-to-handle samples: slurries, sludges, pastes, viscous materials such as glue, tar, or RTV sealant, metal films and machine parts, etc. The 3571 X-CELL®has a body open at both ends, two snap-on rings, and a collar to hold film in place while the ring is being applied. After window film has been attached to one end of the cell, it can be inverted, and the sample placed, poured, dabbed, or otherwise located in the cell, directly against the window. The top of the 3571 X-CELL®can then be left open or sealed with film.

If you deal with small air-sensitive or hazardous samples, then the 3577 Micro X-CELL®is the solution to your problems. While it fits in the standard 31 mm sample holder, the window is 6.3 mm in diameter, and only 0.5 mL fills it up. Collimate your beam down to reduce scatter and you're ready to go. The Micro X-CELL®can be run with an open or window back, but it will also accept a 7 x 13 mm serum bottle closure for injection, purging, or the isolation of special samples.

BUBBLE-FREE CELL INSERT

The Bubble-Free Cell Insert is a simple insert for the 40 mm XRF liquid cell (3527) which prevents trapped air from forming a bubble under the window film when the cell is used in the upright position. The 3527I Bubble-Free Cell Insert is pushed into a cell before the sample is added, and forms an inverted-cone shelf under which air bubbles can be trapped once the cell is filled and sealed. In many XRF systems, liquid cells are run in an inverted position, so any bubbles rise to the back of the cell and do not interfere with the analysis. The drawback of this "inverted optics" configuration is that if the window film ruptures, the sample can spill and potentially contaminate or damage the spectrometer. However, an increasing number of samples are being run in so-called "normal optics" XRF spectrometers, where the X-ray source is above the cell instead of underneath. Bubbles trapped during assembly of the cell can collect against the window, displacing the sample and attenuating the X-rays reaching the sample and emitted by it. To use an X-CELL®fitted with a SPEX SamplePrep Bubble-Free Cell Insert, assemble the cell, invert it, and tap it. Any bubbles should rise through the hole in the insert, and when the cell is turned right-side-up the bubbles will be trapped below the insert's shelf, away from the window film. Most but not all bubbles get into an X-CELL®during assembly; some samples outgas when heated by the X-ray beam. Tests have shown that a pinhole near the edge of the window film will allow such

gases to escape without bulging or rupturing the film.The 3527I Bubble-Free Cell Insert coupled with the 3527 X-CELL®will fit normal optics XRF spectrometers made by Rigaku and Shimadzu Scientific Instruments, Inc.

SELECTING THIN FILM WINDOWS FOR XRF

An X-CELL®liquid sample cell must be covered by a thin film which serves as the sample window. The film must effectively contain the liquid while allowing uniform transmission of the X-rays to the sample. Your choice of a window film depends on your analytical priorities and the nature of the sample. Is strength more important than X-ray transmission, or vice versa? Are there certain impurities you cannot tolerate in the film? Is the sample chemically aggressive, capable of weakening or penetrating certain films? Is it hot when you pour it into the X-CELLS® , or will it heat up during analysis and perhaps soften the film? Will it release volatiles and put pressure on the film from inside the cell?

Often the final choice of film balances several requirements. We recommend comparing films, and for that purpose we can supply on request samples of SPEX films and X-CELLS®. We also suggest strongly that if there is any question about the compatibility of a sample with certain films and/or X-CELLS® you test them together outside the spectrometer.

WHEN CHOOSING A THIN FILM WINDOW FOR YOUR APPLICATION, CONSIDER THE FOLLOWING FILM CHARACTERISTICS:

1. **Strength:** Leakage or rupture of the window during a run could cause damage to spectrometers with inverted optics. While SPEX SamplePrep accepts no responsibility for damage resulting from window leakage, the films offered by SPEX SamplePrep have been selected for their strength and durability. Kapton is the closest thing to an indestructible XRF window film. It is much stronger than any other SPEX SamplePrep film, heat-resistant to over 300 °C, and chemically impervious to almost everything except strong alkali solutions. Six micron (6 µm) Mylar® is also unusually strong for its thickness. The remaining films (5 µm Polypropylene, 4 µm Ultralene®, and 3 µm Mylar®) have roughly comparable mechanical strengths, but differ in other key properties. Ultralene® remains our best film in its balance of X-ray transmission, chemical resistivity, strength, and purity. All SPEX SamplePrep XRF window films except Kapton are weakened by heating much above 100 °C.

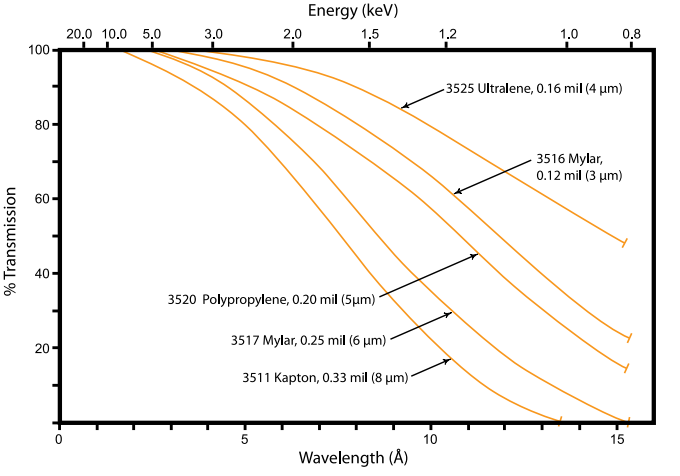
2. **Transmission:** X-ray transmission through polymer films is affected by the thickness of the film as well as its composition. Of the SPEX SamplePrep XRF window films, 8 µm Kapton® is the least transparent to X-rays; 6 µm Mylar® is more transparent, as is 5 µm polypropylene. The thinnest films (4 µm Ultralene, and 3 µm Mylar) have the greatest degree of X-ray transmission, and should be considered for light element analysis in particular.
3. **Uniform Thickness:** The absorption of X-rays at a given wavelength is directly proportional to the thickness of the film. Thus you will get uneven fluorescent intensity with an uneven film. SPEX SamplePrep films are selected for uniform thickness so your results are reproducible.
4. **Purity:** No XRF window film is absolutely free of metallic impurities, but some are cleaner than others. While Mylar film makes a suitable window for most analyses, it may contain trace levels (ppm) of Ca, P, Fe, Cu, Zn, or Sb. Polypropylene film has been known to contain trace levels of Ca, Zr, P, Fe, Zn, Cu, Ti, and Al. Our cleanest film overall is Ultralene. Kapton is clean in most respects but now includes a phosphate-based surface coating to improve its handling characteristics. Our best advice is that you run a blank from every new roll or box you open since impurities can be different not only between types of film but also between different lots of the same material. SPEX SamplePrep cannot be held responsible for variation in film composition.
5. **Chemical Attack:** Your sample's chemical characteristics may dictate your choice of window film. Some samples simply attack some films, exposing the spectrometer to possible damage if the window leaks. For each new sample, we strongly recommend that you test the window film by exposure to the sample for several times your longest anticipated running time. The tables which follow may be of some help in selection, but they are not meant to replace actual testing.

In general, Kapton is highly resistant to acids and organic chemicals and almost everything except strong bases. Mylar is vulnerable to strong acids and strong bases and resistant to organic chemicals. Polypropylene and Ultralene® are reasonably resistant to acids, bases, and many organic chemicals, but can be attacked by aromatic and halogenated hydrocarbons. All of these films are attacked by strong oxidizing agents.

X-CELLS® are made of polyethylene, which is generally resistant to acids, bases, and most organic chemicals except halogenated hydrocarbons. Window films, being much thinner than X-CELL®bodies, are likely to perforate long before the X-CELL®is affected. However, polyethylene begins softening at temperatures well below 100 °C, so if you are working with hot samples, pre-test the X-CELL®body as well as the window film.

CAUTION: SPEX SamplePrep window films and X-CELLS® are not intended to be used in vacuum-path XRF systems. An abrupt pressure change in an XRF spectrometer's sample chamber can stretch or burst window film, or otherwise cause the X-CELL®to leak or come apart. It should also be noted that some volatile chemicals have the ability to generate pressure inside an X-CELLS®, and can stretch a film to bursting or (rarely) cause slow leakage by migrating through the joint between the film and the cell. Whenever there is a question about whether a SPEX SamplePrep X-CELLS®, window film, or combination thereof is likely to perform properly in a particular analytical situation, the analyst should first test these products in a way which does not risk contaminating the spectrometer.

THIN FILM TRANSMISSION CHARACTERISTICS



SAMPLE GRIND / FUSION / PRESSED PELLET REQUEST FORM

Send samples to SPEX SamplePrep at 65 Liberty Street, Metuchen NJ 08840, USA. Fax form to +1 (732) 906-2492 or fill out the form online at www.spexsampleprep.com.

Please contact us before sending in your test sample. Call (732) 623-0465: or email sampleprep@spex.com. Samples are required to be sent on customers account, excess sample that is considered to be hazardous will be returned. Up to three samples will be ground at no charge. A report and recommendations will be returned within three weeks.

Select a SPEX SamplePrep product to demo, or receive a quote:

- ☐ 2010 Geno/Grinder®
- ☐ 1200 GenoLyte®
- ☐ 1600 MiniG®
- ☐ 6775 Freezer/Mill®
- ☐ 6875 Freezer/Mill®
- ☐ 6875D Freezer/Mill®
- ☐ 8000M Mixer/Mill®
- ☐ 8000D Mixer/Mill®
- ☐ 3636 X-Press®
- ☐ 8530 ShatterBox®
- ☐ K1 Prime
- ☐ X-300 X-Fluxer®
- ☐ X-600 X-Fluxer®

SPEX SamplePrep grinding or mixing container _____

Sample description and composition _____

If sample is incompatible with any grinding container or considered hazardous in any way please include MSDS.

Purpose for Grinding _____

Emission Spectroscopy _____ X-Ray Spectroscopy _____

Extraction _____ Other _____

Desired Particle Size after Grinding, if known _____

If contamination with certain metals or plastics is objectionable, please list them along with the approximate ppm level(s) of concern _____

Notes or Comments _____

For Return of Sample and Report

Name and Title _____

Company Name _____

Mailing Address _____

Phone _____ Date mailed _____

Shipping Account number _____

TRADEMARK REFERENCES

REGISTERED TRADEMARKS OF SPEX SAMPLEPREP, LLC

- Auto-Extractor®
- Bench-Press®
- Canna-Prep®
- Freezer/Mill®
- Geno/Grinder®
- GenoLyte®
- Katanax®
- Kryo-Tech®
- MiniG®
- Mixer/Mill®
- PrepAid®
- Spec-Cap®
- ShatterBox®
- SPEX®
- Ultralene®
- Ultrabind®
- X-Press®
- X-Cell®

REGISTERED TRADEMARK OF INTERNATIONAL CENTRE FOR DIFFRACTION DATA®:

ICDD®

REGISTERED TRADEMARK OF APPLIED BIOSYSTEMS:

TaqMan®

REGISTERED TRADEMARKS OF E.I. DUPONT DE NEMOURS & COMPANY:

- Delrin®
- Kapton®
- Teflon®
- Viton®
- Vertrel XF®

REGISTERED TRADEMARK OF DUPONT TEIJIN FILMS:

Mylar®

REGISTERED TRADEMARK OF KATANAX INC:

- Katanax®
- X-Fluxer®

REGISTERED TRADEMARKS OF QIAGEN:

- QIAshredder®
- DNeasy®

REGISTERED TRADEMARK OF ROCHE MOLECULAR SYSTEMS AND HOFFMAN LAROCHE:

PCR®

DEMO REQUEST/ QUOTE FORM

First Name _____

Last Name _____

Title _____

Company Name _____

Address _____

Address 2 _____

City _____ State/Province/Region _____

Country _____ Zip/Postal Code _____

Phone _____ Email _____

Select a SPEX SamplePrep product to demo, or receive a quote:

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- ☐ 6875 Freezer/Mill®
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- ☐ 8000D Mixer/Mill®
- ☐ 3636 X-Press®
- ☐ 8530 ShatterBox®
- ☐ K1 Prime
- ☐ X-300 X-Fluxer®
- ☐ X-600 X-Fluxer®

Fax form to +1 (732) 906-2492 or fill out the form online at www.spexsampleprep.com.
Demo Equipment is required to be sent on the customer's shipping account.

ORDERING INFORMATION

PRICES AND QUOTATIONS

Prices are subject to change without notice. We will gladly quote current prices on request. Verbal and written quotes are valid for 30 days unless otherwise stated. Acceptance by the seller is expressly limited to the Standard Terms of SPEX SamplePrep, LLC.

CONFIRMING ORDERS

Mark confirming orders clearly. Failure to do so will result in a restocking charge for duplicate shipments.

DOMESTIC ORDERS (US)

PAYMENT

Payment terms are net 30, days based on credit approval. If you do not have an established account with SPEX SamplePrep, LLC, you may provide credit information, three commercial references, Dun and Bradstreet Number, or remit payment including freight charges with order. Payment may also be made by credit card (Visa, MasterCard or American Express), or wire transfer.

SHIPPING

Our shipping terms are EXW/FCA Metuchen, NJ. Any other terms are by special arrangement only. Most items are shipped by UPS or motor freight, depending on the size and weight of the shipment. Various express/overnight services are also available. Shipping charges can be pre-paid and the cost will be added to the invoice. If you are sending payment with the order, consult us first for shipping charges.

MINIMUM ORDERS

The minimum order is $100. Replacement parts and repair charges are excluded from this minimum.

EXPORT ORDERS

SPEX SamplePrep maintains authorized dealers in many countries around the world. In Europe we are represented by our subsidiary, SPEX CertiPrep, Ltd. To find a distributor in your country please visit our website or contact our main office via email at sampleprep@spex.com. Orders from countries without authorized dealers may be placed directly through SPEX SamplePrep. However, export ordering terms, instructions, and quotations must be obtained in advance. Orders received without prior agreement may be returned to the purchaser.

PAYMENT

Prepayment of merchandise cost and shipping charges is required for all export orders. Payment may be made by check, wire transfer, letter of credit, or credit card (Visa, MasterCard, American Express). Minimum order for letter of credit payments is $2,500.00. All banking charges must be to buyer's account. Any letter of credit that states "banking charges are for the account of the beneficiary" WILL NOT BE ACCEPTED.

WARRANTY

SPEX SamplePrep LLC guarantees its products against defects of materials and workmanship for one year from the date of original shipment. Repairs and replacements made under warranty are guaranteed for the remaining original warranty period. The warranty excludes wear parts: that wear out through use and have to be replaced periodically for proper operation. Wear parts include not only gaskets, drive belts, grinding media, and the like, but also pneumatic cylinders, the Geno/Grinder ball slide, and the Mixer/Mill clamp retaining spring assembly. Also excluded from the warranty are grinding vials and containers made from tungsten carbide, alumina ceramic, zirconia ceramic, silicon nitride, and agate. The customer pays return freight for warranty claims, but SPEX SamplePrep will pay return freight to the customer if the warranty claim is valid. SPEX SamplePrep reserves the right to judge whether a malfunction is due to defects in materials or workmanship, or to wear, negligence or misuse. All Katanax instruments have been carefully inspected and tested before shipping and are warranted to be free from defects in workmanship and material for a period of one year from date of shipment. Hard ceramic parts may exhibit small cracks developed under heat, and will not be considered defective unless this situation impairs functionality of the instruments. Heating elements are warranted for a period of six (6) months.

TO ARRANGE A RETURN SHIPMENT

We want you to be happy with your purchase from SPEX SamplePrep. Please bring any problem to our attention, but DO NOT RETURN any item before contacting us for a RMA (Returned Materials Authorization) number, and instructions. Unauthorized returns will be refused. Cost for all return transportation is the responsibility of the customer. Credit for returned merchandise is issued only after the goods have been received and inspected, and returns are subject to a restocking fee. Address all shipments to SPEX SamplePrep, LLC, 65 Liberty St., Metuchen NJ 08840; always include the RMA number.

SERVICE

The majority of our products are designed and built in our factory in Metuchen, New Jersey. The SPEX SamplePrep Service Department is an integral part of our operation, and can be counted on to maintain, upgrade, repair, or rebuild SPEX SamplePrep equipment with factory parts and the necessary expertise. Our trained service technicians can also advise you on the most likely cause of a problem. Repairs are subject to a minimum two-hour labor charge, and prices are subject to change without notice. Out-of-warranty repairs and parts are guaranteed for 30 days. Before returning equipment for service, contact our technicians for an RMA number and cost estimate, and include a purchase order with the return. Address returns to our 65 Liberty St., Metuchen, NJ 08840 sampleprep@spex.com. The customer pays freight both ways for equipment to be serviced.

PRODUCT SPECIFICATIONS

Every effort has been made to provide complete and accurate product information in this catalog. However, specifications are subject to change without notice, and changes may be made from time to time to improve the performance of our products.

PRECAUTIONS

SPEX SamplePrep products are not for any household application. Our acceptance of a purchase order is made with the assumption that our products will be used only by qualified individuals who are trained in appropriate procedures. Users are responsible for knowledge and understanding of the potential hazards of the material with which they are working. SPEX SamplePrep does not recommend unattended operation of any of our laboratory equipment.

SUBJECT INDEX

PRODUCT INDEX

NOTES

NOTES

SPEX SamplePrep has been providing superior sample preparation equipment and supplies since 1954. This handbook, now in its 14th edition, has been an important tool that provides our customers not only with information about our products, but also how they can be used in different techniques and applications. This is backed by our unique knowledge and expertise to help our customers select the most appropriate products for their sample preparation needs.



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