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3636
X-Press®

3636 X-PRESS®

The 3636 X-Press® is a 35-ton (31.8 metric ton) hydraulic laboratory pellet press that accepts most standard die sets. This automated laboratory press is created to press sample pellets for spectroscopy. It is programmable, so that repetitive pellet preparations may be carried out with minimum operator workload and a maximum of uniformity. The X-Press can also be operated manually.

APPLICATIONS

Pressing sample pellets for XRF/IR

SAMPLE TYPES

- Pharmaceuticals
- Cement
- Minerals
- Rocks
- Slag
- Soil

Demo and Sample Test Programs are available for this product. See pages 110-111 for more details

3636 X-Press®

Part number:
3636-115 & 3636-230

For video, scan with your smart phone or visit www.youtube.com/sampleprep

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.

FEATURES & SPECIFICATIONS

1. Safety door and interlock with automatic pump shut-off safety valve.
2. Adjustment screw secures the loaded sample die placed on top of the platen.
3. Accepts all standard die sets.
4. 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.
5. Handle and handwheel moves the adjustment screw to secure the sample.
6. Access panel can be removed to fill the oil reservoir or check the oil level.
7. Manual pressure relief valve used to release the hydraulic system pressure in an emergency or for system maintenance.

Voltage	115V/60Hz or 230V/50Hz	Daylight	Adjustable 2.0 to 6.0 inches (5 to 15 cm)
Weight	145 lbs. net (66 kg)	Motor	1/3 hp
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.)	Dimensions	20.0 in. (50.8 cm) x 13.0 in. (33 cm) x 22.5 in. (57.2 cm)
Power Cord	3-prong grounded plug 115V, 60Hz. 2-prong European cord for 230V, 50 Hz		

SPEX CARVER MANUAL PRESSES

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 Approved.

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 Approved.

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 Approved.

X-PRESS ACCESSORIES

PELLET DIE SETS

SPEX SamplePrep dies are designed to fit all SPEX SamplePrep presses, and they are equally compatible with other standard laboratory presses using stand-alone dies.

3610 Die Set – 10 mm
Includes evacuable die body, base, plunger, two 10 mm polished steel pellets, O-ring vacuum seals, knock-out ring for sample disk extraction. Load limit 5 tons (4.5 metric tons).

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 40mm 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 92 and 93 for details about SPEC-Caps and PrepAid Binders

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 beginning in Section 2, Chapter 4), 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 full range of laboratory presses, pellet die sets, and die accessories. Presses include a 12-ton and 25-ton SPEX SamplePrep-Carver manual presses and the 35-ton 3636 Automated X-Press. Pellet dies are available in the standard sizes of 10 mm,13 mm, 31 mm, 35 mm, and 40 mm. Accessories include: aluminum Spec-Caps to form and protect 31 mm, 35 mm, and 40 mm pellets; cellulose and paraffin Prep-Aid binders, as well as SPEX SamplePrep's own UltraBind; and Sleeve-and-Plunger sets to form sample pellets with a thin layer of sample on a binder matrix.

While methods and mills for pulverizing are as varied as the samples themselves, pellet pressing requires simpler tools. SPEX SamplePrep presses, dies, and pelletizing accessories are sophisticated in design, but functionally simple and very effective.

Reproducibility is a cardinal virtue in sample preparation. SPEX SamplePrep dies and presses ensure production of uniform sample disks, whatever the sample or analytical technique.

PREPARING POWDER SAMPLES FOR XRF

X-ray fluorescence spectrometry often requires the sample to be in a homogeneous powdered form with a planar surface. Although this can be accomplished by spreading the powder on the window film of a cell designed for liquid samples (such as a SPEX SamplePrep X-Cell), compression of the powder in a pellet die yields a denser, flatter surface that provides greater analytical accuracy and sensitivity, especially for wave length 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.

In the end, an exact procedure must be developed for each type of sample. The sampling method and the amount of sample to be ground, the type of mill and the grinding time, what size of die and what pressure to use, whether to include a binder or press the disk in a Spec-Cap, all these details may vary and must be worked out by the analyst to suit his or her samples, equipment, and analytical requirements. Running a series of standards or known samples will help to confirm that your chosen procedure results in accurate, reproducible data.

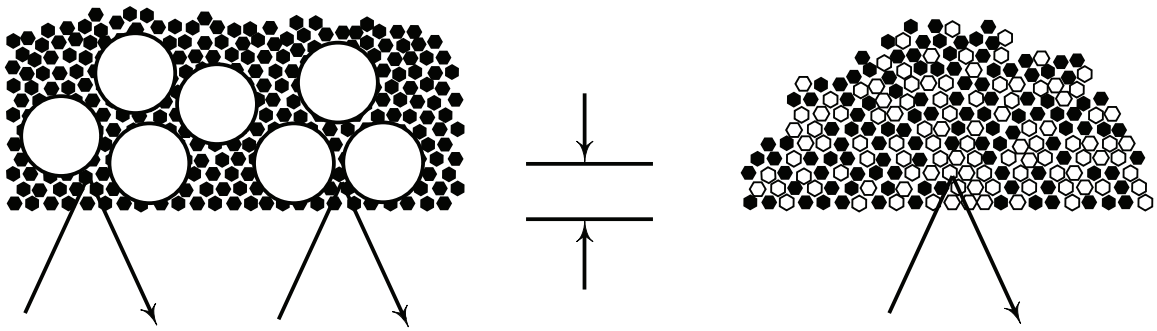
PARTICLE SIZE EFFECTS

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.

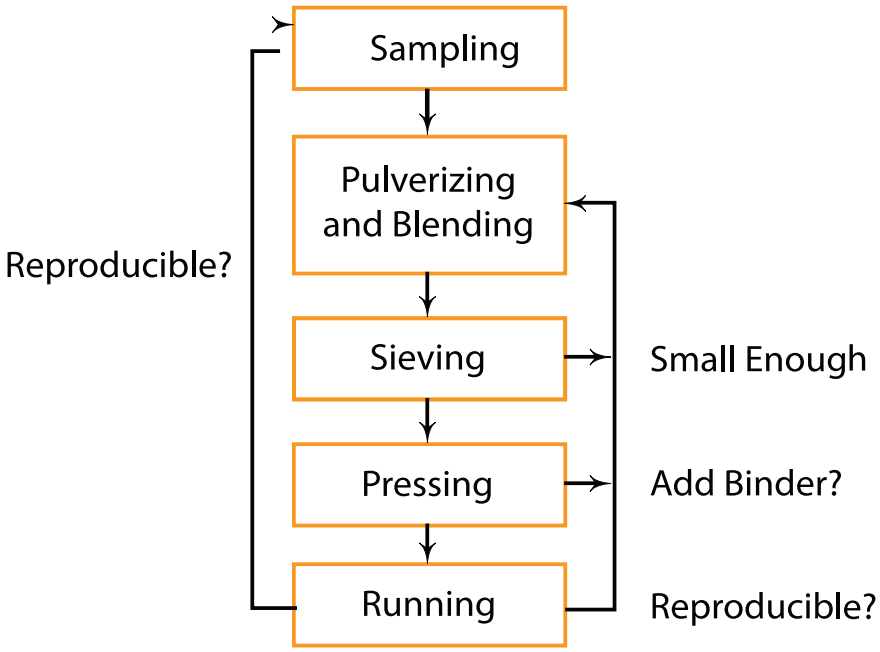
As an X-ray enters a pelletized sample disk the exciting radiation is absorbed by the matrix. When a particle within the matrix absorbs radiation, the resulting fluorescence is also absorbed, in whole or in part, by the matrix. Hence there is a limit to the depth to which the existing radiation can penetrate 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 on the next page. 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.

Pressing and Pelletizing



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



The correct particle size is often determined empirically

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. SPEX SamplePrep offers laboratory mills and grinding vials that are ideal for this procedure.

PRESSING SAMPLE DISKS

To make a sample disk, you need a sufficient quantity of homogeneous powder, often a mixture of thoroughly pulverized sample material and an appropriate binder; a pellet die set, including two pellets of polished steel or other hard, smooth material for sandwiching the sample powder; and a machine for exerting the necessary pressure on the powder. An optional aluminum cup such as the Spec-Cap contains the powder, protects the die bore against abrasive samples during pressing, and subsequently protects the pellet against chipping or breaking. An alternate technique, which yields a durable disk of solid binder with a thin layer of sample on one side, involves the use of a sleeve-and-plunger set with a pellet die.

The powder to be pressed must be fine-grained and homogeneous, in any case. If the pulverized sample material is not readily compressed into a stable, cohesive disk, it may be necessary to add a binding/ briquetting agent such as cellulose, paraffin, or UltraBind. A binder can be uniformly blended with the sample or used as a backing for a thin layer of sample. Binders when properly used enhance analytical accuracy by stabilizing the sample disk and keeping it from distorting or crumbling during analysis and/ or storage.

In an ideal sample disk pressed for XRF, the analytical surface of the disk is not only flat but also uniform, and truly representative of the original sample. This is true whether the sample was pressed without binder in a Spec-Cap, blended with a binder before pressing, or pressed in a thin layer on a binder matrix. Compact, flat, stable, and uniform sample disks can be ensured by the use of reliable SPEX SamplePrep dies, presses, accessories, and Prep-Aid binders.

While the pressing of powders into disks for XRF analysis is, in all its details, a fairly intricate exercise, it can be routinized to yield consistent, reliable analytical results. A review follows of the processes and tools involved in the actual pressing (pellet dies, binders, and anti-sticking agents, etc.). The discussion assumes that the analyte has been properly sampled (so that it is representative) and adequately pulverized (to minimize particle size effects).

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. Manual and motorized hydraulic presses are available for this task: SPEX SamplePrep offers 12-ton and 25-ton SPEX SamplePrep-Carver manual presses and the 35-ton 3636 Automated X-Press. Any SPEX SamplePrep pellet die will fit these presses.

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 3636 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 3636 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. Each has its proper application and technique, detailed later in this chapter.

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 diebore, 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-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 palletized, 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 200 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 a 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. Graphite was used extensively in the development of sample-disk pressing techniques, but that was because of its high conductivity. The 31 mm sample disk accepted as the standard size by most XRF manufacturers and users was originated for the point-to-plane technique in arc/spark optical emission spectroscopy, where a high-voltage current is passed through the sample. Sample disks pressed with graphite are also used for spark ablation techniques.

Solid binders also include powdered wax, pulverized acrylic plastic, and others. Liquid binders are also used; they are blended with a sample prior to pressing. Most appear to be a solution of polymer(s) in solvent(s); their reputed advantage is that during blending, all the sample particles receive a light coating of the polymer, so that subsequent pelletizing is more direct and efficient.

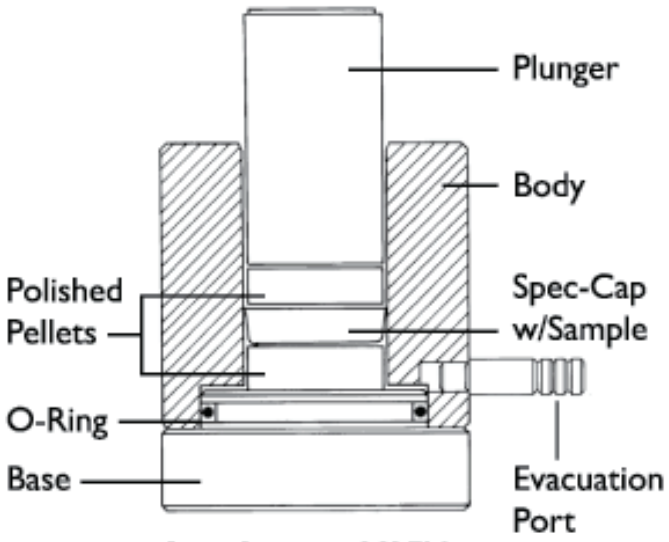
There are also materials which are touted as aids for both grinding and binding. Generally these are in tablet form, so that a uniform amount can be easily added to the sample just before grinding. The classic example of this genre is aspirin, but there are more sophisticated, proprietary grinding/binding tablets available. Of the use of binders in general, it should be remarked that their most conspicuous property is that of caking under pressure, or at least of flowing under pressure and then caking 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. SPEX SamplePrep offers dies to make four standard sizes of sample disks: 13 mm for IR spectrometers, 31 mm for most OES and XRF instruments, and 35 mm and 40 mm for newer XRF spectrometers. Dies are clearly labeled with bore size and maximum safe load.

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.