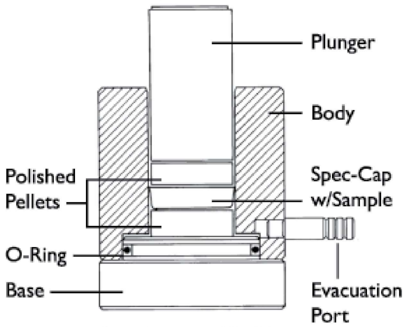
cement-mix ground to below 10 microns) is more likely to bind well under pressure than the same sample ground to moderate fineness, and that grinding aids are often needed to attain very fine particle size. When a grinding aid such as Vertrel® XF (3650) is used, the binder can usually be added to the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

Katanax

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.

Katanax

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.

Katanax

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

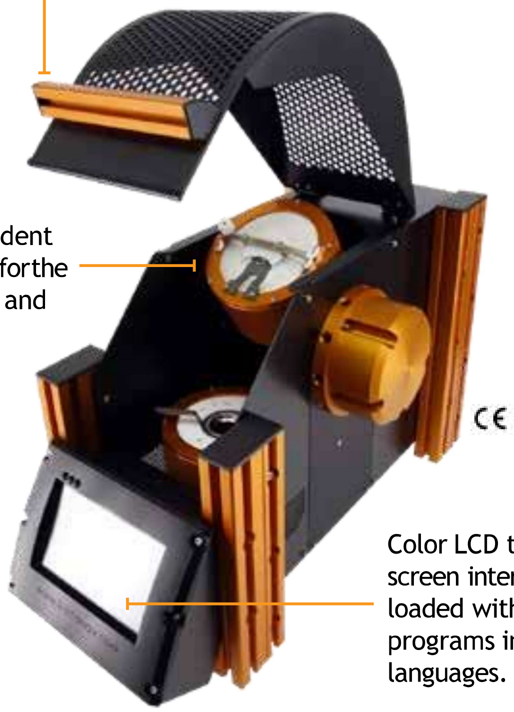
VOLTAGE	195-250 VAC (50-60 Hz)
DIMENSIONS	56 cm (22 in.) x 105 cm (41 in.) x 69 cm (27 in.)
MAXIMUM POWER	4,000 W
WEIGHT (LBS)	187 lbs. (85kg)
OPERATING TEMPERATURE	20–1,200 °C
FREQUENCY	50-60 Hz
BREAKER	Built in 20 amps

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.



Katanax

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

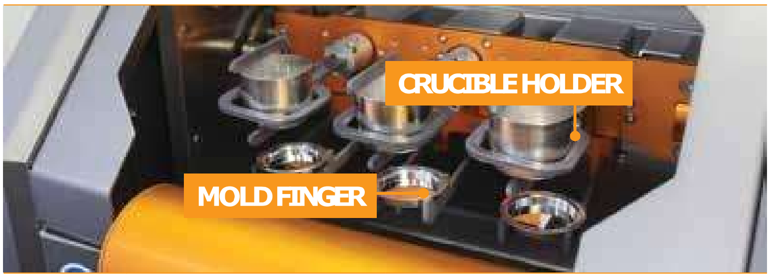
KP7010A - TEFLON® BEAKER
Square PTFE (Teflon) beaker with magnetic bar, X-600

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



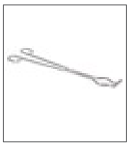
KP8500A Spare kit basic

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

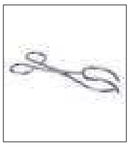
KP0010A BEAKER PTFE
(Teflon) with magnetic bar, X-300

Katanax

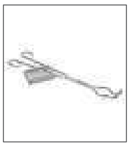
HANDLING TOOLS FOR USE WITH MUFFLE FURNACES



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.

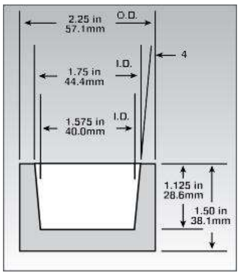
7158 GRAPHITE CRUCIBLES
Regular-purity graphite crucible also capable of casting 31 mm glass disks. 44.4 mm O.D. x 38.1 mm tall. 31 mm I.D. with flat bottom. 27 mL capacity. Sold in packs of 10

7159 GRAPHITE CRUCIBLES

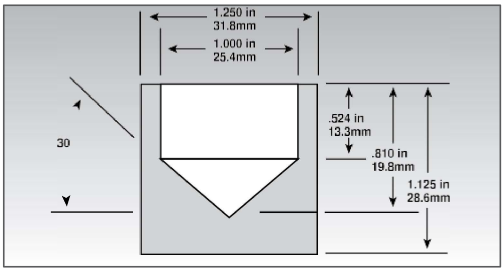
High-purity graphite crucible also capable of casting 33.7 mm glass disks. 50.8mm O.D. x 38.1 mm tall. 33.7 mm I.D. with flat bottom. 32 mL capacity. Sold in packs of 10

7161 GRAPHITE CRUCIBLES

For casting 40 mm glass disks, 57.1 mm O.D. x 38.1 mm, 44.5 mL capacity, 40mm 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.](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 TiO_2, 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.