### Katanax®



**Automatic fluxer** 

Thank you for having chosen the K2 Prime fusion machine from Katanax. To enjoy years of reliable, efficient and safe use of this time-saving instrument, please read this manual thoroughly and keep it in a safe and handy place for future reference.

Should you have any question regarding the use, maintenance or repair of your instrument, kindly contact Katanax directly for assistance (see page 91 for contact details).

© February 2015, Katanax inc. All rights reserved.

### Index

| Inc | ndex                                  | 2  |
|-----|---------------------------------------|----|
| Ins | nstallation                           | 5  |
|     | Box contents                          |    |
|     | Location                              |    |
|     | Leveling                              |    |
|     | Heating element protection            |    |
|     | Hi-throughput "HX" version            |    |
|     | Connection                            |    |
|     | Questions ?                           |    |
| Tni | ntroduction                           | 9  |
|     | The fusion theory                     |    |
|     | Automatic fluxers                     |    |
|     | The K2 Prime fluxer                   |    |
|     | Main features of the Katanax K2 Prime | 12 |
| _   |                                       |    |
| Pre | recautions                            | 14 |
| Но  | ow the unit works                     |    |
|     | Heating                               |    |
|     | Access to the furnace                 |    |
|     | Agitation and pouring of the melt     | 15 |
|     | Cooling                               | 16 |
|     | Solution agitation                    |    |
| Us  | sing the K2 Prime (basic)             | 17 |
|     | Operation of the safety shield        | 17 |
|     | The main running screen               |    |
|     | Electro-mechanical pushbuttons        | 20 |
|     | Loading a program                     |    |
|     | During a fusion                       |    |
|     | The right ingredients                 |    |
|     | Care of the elements                  |    |
|     | Care of the platinumware              |    |
|     | Crucible installation                 |    |
|     | Mold installation                     |    |
|     | A general fusion                      |    |
|     |                                       |    |
|     | Making solutions                      |    |
|     | Description of the fusion steps       |    |
| Pro | rogramming the K2 Prime (advanced)    | 33 |
|     | Viewing the fusion parameters         |    |
|     | Unlocking the advanced mode           |    |
|     | Managing fusion methods               |    |
|     | Preparing a fusion program            |    |
|     | Heating steps                         | 39 |
|     | Pouring sten                          | 41 |

|     | Cooling steps  | 42   |
|-----|--|--|
|     | On-the-fly editing   | 43   |
| Gla | bal parameters   | 11   |
| GIU | Language   |  |
|     | Holding temperature offset   | 44<br>44   |
|     | Startup tolerance  |  |
|     | End beep   |  |
|     | Automatic shut-off delay   | 45<br>45   |
|     | Safety shield protection   |  |
|     | Fusion counter   |  |
|     |  |  |
| Spe | cial parameters  | 47   |
|     | Accessing the Special parameters   | 47   |
|     | Twisting correction  | 47   |
|     | Offsets  | 48   |
|     | Cam assisted   | 49   |
|     | Movement testing   | 49   |
|     | :  |  |
| Fus | ion troubleshooting  |  |
|     | Disk cracks  |  |
|     | Disk crystallizes  | 51   |
|     | Incomplete disk  | 52   |
|     | Non-homogenous disk  |  |
|     | Bubbles in disk  | 52   |
| Per | iodic inspection   | 53   |
|     | Warning  |  |
|     | Inspection schedule table  | 53   |
|     | Flux spillage on holders   | 54   |
|     | Holders alignment and functionality  |  |
|     | Visual integrity of heating elements   |  |
|     | Element terminal connections   |  |
|     | Door springs tension check   |  |
|     | Furnace cleanliness  |  |
|     | Thermocouple junction  |  |
|     | Door Safety switch   | 58   |
|     | Linear motion system   |  |
| _   | ·  |  |
| Ser | vice operations  |  |
|     | Warning  |  |
|     | Platinumware holders service   |  |
|     | Crucible holder removal  | 60   |
|     | Crudble Holder Terrioval   | 62   |
|     | Crucible holder assembly   |  |
|     | Crucible holder assembly   | 63   |
|     | Crucible holder assembly   | 63<br>64   |
|     | Crucible holder assembly   | 63<br>64<br>67                                     |
|     | Crucible holder assembly Crucible holder Installation Mold holder removal Mold holder assembly Mold holder installation  | 63<br>64<br>67<br>68                               |
|     | Crucible holder assembly Crucible holder Installation Mold holder removal Mold holder assembly Mold holder installation Adjustment of holders  | 63<br>64<br>67<br>68<br>68                         |
|     | Crucible holder assembly Crucible holder Installation Mold holder removal Mold holder assembly Mold holder installation Adjustment of holders Heating element replacement  | 63<br>64<br>67<br>68<br>68<br>77                   |
|     | Crucible holder assembly Crucible holder Installation Mold holder removal Mold holder assembly Mold holder installation Adjustment of holders Heating element replacement Element tube replacement   | 63<br>64<br>67<br>68<br>68<br>77<br>79             |
|     | Crucible holder assembly Crucible holder Installation Mold holder removal Mold holder assembly Mold holder installation Adjustment of holders Heating element replacement Element tube replacement Furnace top/back removal                            | 63<br>64<br>67<br>68<br>68<br>77<br>79<br>80       |
|     | Crucible holder assembly Crucible holder Installation Mold holder removal Mold holder assembly Mold holder installation Adjustment of holders Heating element replacement Element tube replacement Furnace top/back removal Spring position adjustment | 63<br>64<br>67<br>68<br>68<br>77<br>79<br>80<br>81 |
|     | Crucible holder assembly Crucible holder Installation Mold holder removal Mold holder assembly Mold holder installation Adjustment of holders Heating element replacement Element tube replacement Furnace top/back removal                            | 63<br>64<br>67<br>68<br>68<br>77<br>79<br>80<br>81 |

| waitancy                                     |  |
|--|--|
| Warranty                                     |  |
| Physical                                     |  |
| Electrical                                   |  |
| Technical specifications                     |  |
| Backup or restore by USB                     |  |
| Padan as sastas by UCD                       |  |
| Preset programs                              |  |
| Firmware and program transfers               |  |
| Linear carriage lubrication                  |  |
| Base compartment access                      |  |
|  |  |
| Furnace backwards flippingBlower box removal |  |

### Installation

Congratulations on your acquisition of the K2 Prime fluxer, from Katanax. Please read the following section for proper commissioning of your instrument. Do not hesitate to contact Katanax with any question you might have with this crucial step.

**IMPORTANT**: It is advisable that at least two persons carry this instrument to avoid injuries. Do not drop instrument.

### **Box contents**

The instrument comes with its essential accessories. In addition to optional items you might have ordered, the box should contain:

- 1 fluxer (K2 Prime main unit), #K2Pn-dd [n=5 or 6 positions, dd=mold diam.]
- 1 accessories box containing the parts to assemble the platinumware holders
- 1 instruction manual (this booklet) and other instructions

Additionally, if you have ordered a K2 Prime with solution-making capability, you will find:

- 5 unbreakable PTFE beakers, 5x#KP0010A
- 5 magnetic stirring bars (included with KP0010A)
- 1 crucible holder middle section (to hold 5 crucibles)

Note that the rest of the solution-making devices are permanently installed into the instrument, and are not packaged separately.

### Location

#### Vent hood

Molten flux, additives and some samples may produce vapors and gases that need to be extracted. In particular, the use of excessive amounts of halogen-based non-wetting agent will potentially cause the deterioration of the fluxer if the corrosive fumes are not properly vented out.

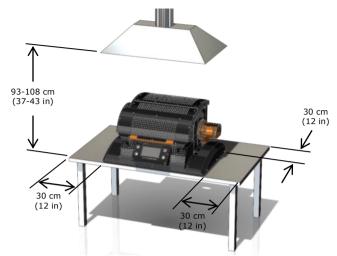
In order to minimize airflow around the unit, the hood shall ideally be of the canopy type, i.e. with sides and front open. If the sides are closed or otherwise occupied by other instruments, a clearance of 30 cm shall be maintained on either sides and to the back of the instrument.

Sash-type hoods should preferably not be used; or if unavoidable, the sash shall be kept open, so as not to create front-to-back air movements.

The funnel (intake section) of the hood shall be approximately equal (or slightly larger than) the size of the instrument's footprint, and centered above it to avoid sideways airflow around the fluxer.

The lowest part of the funnel shall sit 45 to 60 cm above the instrument (93 to 108 cm above the tabletop).

Fume hood air speed, measured at the base of the intake funnel, shall be around  $0.5 \, \text{m/s}$ .



Given the nature of the exhausted gases (halogen compounds), Katanax recommends that the piping be internally coated with a corrosion-resistant finish, PTFE-coated piping being the optimal. (Other corrosive-resistant coatings may be allowed by your local fire codes.) The same applies to the fan / impeller and any other part in contact with the gas flow.

Motor and fan / impeller sizing information can unfortunately not be provided by Katanax, as those strongly depend on factors linked to each individual installation (pipe diameter, pipe distance to the outside, exhaust baffle type, etc...). Those will need to be calculated locally for each installation by HVAC engineers.

In all cases, the vent hood must be centered above the fluxer. Failure to comply with this requirement could create temperature uniformity issues and/or premature degradation of the platinumware holders.

#### **Table**

The counter on which the instrument is to be used must be able to withstand a weight of 68 kg (150 lbs). It is recommended to use the instrument from a standing position, and adjust table height accordingly.

The tabletop shall be made of a non-combustible material, horizontally flat, rigid, and stable.

It is advisable that at least two persons carry the instrument to avoid back injuries.

#### Safety clearance

Because your new fluxer will produce heat, a minimal safety clearance must be provided to prevent surrounding material from heating and potentially catching fire. All around the instrument, a minimum distance of 30 cm (12") must be free from combustible materials. Similarly, a clearance of at least 45 cm (18") must be provided above the top of the instrument.

### Leveling

In order to obtain glass beads with a regular thickness, it is important that the molds be reasonably horizontal upon pouring. If in doubt, place a lightweight bubble spirit level on the molds and adjust instrument's feet to compensate for possible slant. (See page 29 for mold installation details.)

### Heating element protection

To protect the heating elements, especially during the initial fusion method development phase, different protection devices are available. They represent a simple way of shielding the elements from the sputtering that may occur during a fusion that is not optimally set yet.

Katanax offers a protective plate, made of a dark-colored ceramic, and clear tubes. The plate covers all bottom elements simultaneously, while tubes can be installed over top elements and/or bottom elements (when no plate is installed).

The dark ceramic plates is very resistant to flux and chemicals, and can be used in all types of sample preparation. This is the protection method that Katanax recommends.

Alternatively, two clear tube material choices are available: Quartz and Sapphire. The Quartz tubes (part number KP5010E, sold as a pack of 8) are inexpensive, but while these tubes will normally last very long, they are not the best choice if you plan on using Iodine-based releasing/non-wetting agent in your fusion recipes. Indeed, those compounds (LiI, KI, NH $_4$ I...) will evaporate almost entirely their iodine, which readily attacks quartz (SiO $_2$ ).

Hence, if you are using (or plan on using) an Iodine-based releasing agent, we do not recommend to install quartz tubes; rather, if you want a protection on the elements, use Sapphire tubes or a protective plate.

#### In short:

- 1. If you **do not use iodine**-based non-wetting agent:
  - You can install optional Quartz tubes (p/n KP5010E) or Sapphire tubes (p/n KP5011E) – or
  - You can install an optional protective plate (p/n KP1200A), with or without tubes protecting the top elements – or
  - You can use the instrument without any optional protection
- 2. If you want to use iodine-based non-wetting agents:
  - You cannot use Quartz tubes.
  - b. You can order/install optional Sapphire tubes (p/n KP5011E) or
  - You can install an optional protective plate (p/n KP1200A), with or without Sapphire tubes protecting the top elements – or
  - d. You can use the instrument without this optional protection

Please refer to page 79, *Element tube replacement* for additional instructions on tube installation and replacement.

### Hi-throughput "HX" version

The K2 Prime is available at:

- the original 15A, 3000W rating, or
- at a boosted 20A, 4000W rating (with the addition of factory-installed KP5201A or field-installed KP5200A).

We call this higher-power unit the "HX version". This HX version allows not only a faster heat-up rate, but also boasts a second mold cooling blower. The combined effect of those two features can save up to 20% fusion cycle time.

Additionally, the HX version includes the protective plate for bottom elements.

However, the electrical current drawn by the HX version is higher than the regular version, and so the power line, outlet and breaker must be chosen accordingly.

### Connection

The K2 Prime works on 220-240 VAC, 50/60 Hz. Note that the in-wall power line and corresponding wall breakers or fuses must be designed to carry at least 15 A for the regular version(20 A for the HX version), with GFCI protection.

While it is technically workable to use the instrument at 208 VAC (standard in some areas), heat-up times will be longer than in the recommended voltage range. Katanax offers step-up transformers that can overcome such local limitations.

**IMPORTANT**: This instrument is to be connected to a GFCI (Ground Fault Circuit Interrupt) breaker, which provides necessary protection to the fluxer installation.

### Questions?

Should you have any question regarding the proper installation and start-up of your instrument, please contact Katanax directly (see information on page 91) for assistance.

### Introduction

This section intends to introduce the reader to the fusion technique and to familiarize him or her to the K2 Prime.

### The fusion theory

Fusion is a technique used to prepare inorganic samples, with a view to analyze them by x-ray fluorescence (XRF), inductively coupled plasma (ICP) atomic absorption (AA) or any traditional wet chemistry method. Typical samples include: cements, ores, slag, sediments, soils, rocks, ceramics, pigments, glasses and even metals.

A fusion can produce either a small, homogenous solid glass disk (or "bead") for XRF, or an acid solution for other analytical methods.

The process of fusion as a sample preparation method exhibits many advantages over other methods, as it does not produce mineralogy, grain size or orientation effects and the result is perfectly homogenous.

In sample preparation by fusion, the sample never actually melts. It is merely dissolved into a solvent. This solvent, generally a lithium borate flux, is solid at room temperature and must be molten to dissolve anything. This is the only reason the process requires heat.

Therefore, the peak temperature of a sample preparation by fusion is determined only by the type of flux, not the type of sample.

Katanax does not recommend exceeding 1050°C when using lithium borates.

Katanax does not recommend exceeding **1000°C** when using **sodium** borates.

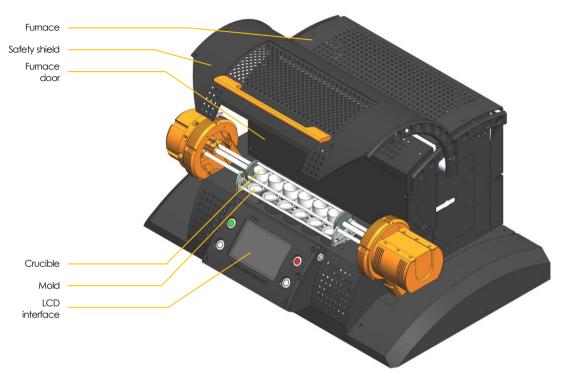
Heating above those temperatures could cause flux evaporation that could bias the subsequent analysis.

### Automatic fluxers

Because of the potential risk of manipulating red-hot samples and to increase repeatability of temperature, mixing and duration, the industry has now adopted the automated fusion machine as the standard equipment to prepare samples by fusion.

### The K2 Prime fluxer

#### General view



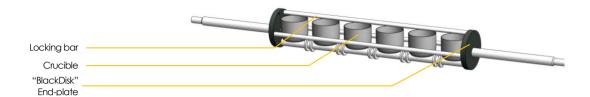
Enter the K2 Prime, the fusion machine that is the evolution of the original K2, which was the first and only fully automated electric fluxer. Based on this success, Katanax has updated the concept, making it sturdier and easier-to-use than ever.

The K2 Prime comes pre-loaded with various fusion methods that can be used as is, or can be customized. All fusion methods can be saved, renamed, deleted or copied, just like computer files. Only the preset methods are protected to avoid accidental overwriting.

Upon turning the instrument on, the furnace will start heating up to prepare for the first fusion cycle. If left idle for an extended period of time, the furnace will automatically turn off. (Holding temperature and automatic shutoff features are further discussed in the *Global parameters* section, at page 44.)

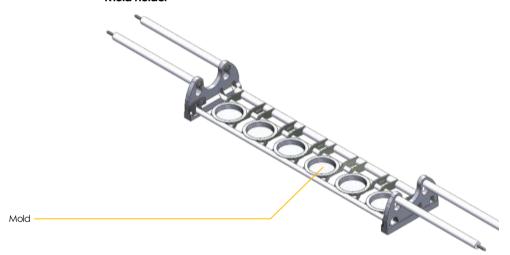
#### Crucible holder

Unlike the first K2 Prime units, this newer version has a fixed locking bar on top of the crucibles. Locking is achieved automatically due to the dynamic geometry during pouring.



The straight-walled platinum crucibles are loaded with a few grams of powdered sample, an appropriate flux and often other agents. The crucibles are inserted into the crucible holder, and held in place by a locking bar.

#### Mold holder



Platinum molds are installed under each crucible in a separate holder. This holder can only accommodate one size of molds at a time.

The crucible holder and the mold holder are referred to collectively as the platinumware holders.

#### Fusion sequence

When the user instructs the K2 Prime to launch the actual fusion, the instrument first checks that the initial temperature is reached (see page 44, *Holding temp* for more details on this setting). If not, the processor waits until the furnace is ready. Then, the furnace door automatically opens, the platinumware holders enter the

furnace, the door closes and all heating steps are automatically started in sequence. Temperature is constantly monitored and displayed.

Once the flux is molten (after a programmable time), a back-and-forth rocking of the crucible holder continuously mixes the flux with the sample.

The liquid flux starts dissolving the sample. When all sample is dissolved (after the programmed time), the furnace door opens, the platinumware holders move forward and, during the time the door closes, the crucibles are tilted forward to empty their contents into the molds.

If set so in the fusion program, it is possible for the now-full molds to be re-heated in the furnace, before they are pulled out again. (This operation is called *annealing* and is needed in very special cases only.)

A blower located underneath cools the molds, while the furnace remains powered, readying for the next cycle. When the molds are completely cooled, the user picks up perfectly homogenous glass beads, ready for analysis by XRF.

For solution preparation, the hot melt is poured into unbreakable beakers (instead of molds), which contain a dilute acid that is automatically stirred by a magnetic system (optional, item no. KP5001A).

Some fusion types, such as peroxide and pyrosulfate, do not even require pouring. The K2 Prime is also designed for such fusions, where the whole crucible is dipped into an acid, after the fusion, to prepare a solution by leaching.

### Main features of the Katanax K2 Prime

### Accuracy

- · Entirely automated
- Fully reproducible fusion methods
- Perfect reproducibility using a closed electric furnace: all crucibles and molds are exactly at the same temperature
- Real-time temperature display

#### Safety

- Integrated locking safety shield
- No gases used, so no post-combustion toxic products released
- Minimal heat dissipation; no need for a powerful vent hood

#### Versatility

- Makes glass disks for XRF and can also readily do peroxide or pyrosulfate fusions
- With optional solution agitation module, can also prepare solutions
- Ready to fuse with built-in methods for oxides, minerals, metals, alloys, sulfides, fluorides and more
- · Can perform solid oxidations
- Fully customizable fusion methods
- USB connectivity

#### **Productivity**

- Simultaneous processing of up to six (6) samples (depending on options)
- Throughput of 20 to 25 samples/hour
- Productivity is improved by a user-adjustable holding temperature. Therefore, the temperature between fusions can be maintained to minimize initial ramping time

### Simplicity

- A simple, intelligent, high-performance furnace
- Easy installation
- Intuitive touch-screen color LCD graphics interface
- Easy icon and menu navigation
- Low maintenance; easy component access
- 1-year limited warranty

### **Precautions**

### **High temperature**

Although this instrument has been built to be very safe, it is still capable of reaching temperatures up to 1200°C. Care must be taken in order to avoid touching hot surfaces.

Even though crucibles and molds are supposed to be cool at the end of a fusion cycle, in order to avoid risks of burns, use appropriate gloves, laboratory tongs or some other adapted tool to manipulate the crucible, mold and glass disk.

User is advised that this instrument remains very hot for a long time, even after turning it off.

#### High voltage

This instrument is powered by 240 Volts AC. Although the elements are interlocked with a safety device that removes power when opening the furnace door, reasonable precautions must be taken.

Disconnect power cord before attempting any cleaning, maintenance or repair operation.

Be careful that no liquid infiltrate into the unit's casing.

### Acid spills

When making solutions, user is strongly discouraged to use glass beakers, as acid spills in instruments are dangerous and not covered by warranty. Use only unbreakable PTFE beakers; otherwise, there is a risk of user injury, due to flying glass shards or acid splatter.

### **Heavy instrument**

It is advisable that at least two persons carry this instrument to avoid injuries. Do not drop instrument.

#### Crucible installation

To ensure safe operation, proper installation of the crucibles needs to be checked by the user before each fusion. See page 27, *Crucible installation*, for detailed instructions.

### Damaged / dirty holders or platinumware

Never run a fusion if platinumware or their holders are damaged or soiled. Replace damaged items or clean dirty parts immediately.

### How the unit works

### Heating

Heating of both the mold and the crucible supports is achieved using a state-ofthe art heating element alloy coiled around rigid ceramic rods. These rods run along a semi-soft, highly insulating material. These parts are then assembled into an aluminum shell.

During heating, temperature is controlled by means of a platinum thermocouple. This same thermocouple also allows the furnace to be kept at a preset, constant temperature to quicken initial ramping before a fusion.

### Access to the furnace

Stepper motors automatically open the door, move the platinumware holders into the furnace, and finally re-close the door. Upon pouring, the same sequence is repeated in reverse order.

During a fusion, the safety shield remains locked, to prevent accidental burns when the platinumware holders move in and out of the furnace.

### Agitation and pouring of the melt

Homogenization of the sample into the flux is produced by the rotation of two synchronous stepper motors located at the far right and left of the instrument. These motors directly drive the crucible holder with a back-and-forth motion to agitate the melt.

Just before the final pouring motion, both motors drive the crucible holders as if to "pour backwards", but the motion is stopped by a mechanical bumper. This ensures perfect alignment of both ends of the crucible holder. (This is called twisting correction.)

At the moment of pouring, the motors rotate the crucible holder forward to an adjustable pouring angle, at an adjustable speed. The melt pours naturally into the molds, and this can be helped with an optional shaking of the crucible in pouring position.

The crucible holder is then automatically brought back to the vertical, ready for another fusion.

The pouring step can also be completely disabled, for procedures such as pyrosulfate or peroxide fusions, which do not need transferring the melt into another container.

### Cooling

When the linear motion system moves the platinumware holder out of the furnace, the molds are stopped just above a cutout designed to receive the beakers.

At a programmed moment, a blower located on the back side of those wells pushes fresh air upwards and under the molds, to cool them.

### Solution agitation

Making solutions requires that the instrument be fitted with the optional solution agitation module, item number KP5001A.

**IMPORTANT:** Before attempting to make solutions, it is necessary to remove the middle section of the mold holder, which can otherwise interfere with the top of the beakers.

When making solutions, the hot melt is poured directly into beakers containing an acid. Those beakers are to be placed into the cooling wells at the beginning of the fusion. This acid solution must be agitated to improve the dissolution speed of the crystallized flux and sample.

To do so, alternatively powered magnetic coils produce a rotating magnetic field under the beakers. By placing a laboratory-type magnetic stirring bar in the acid before fusion, the agitation will be automatically started at the appropriate moment.

## Using the K2 Prime (basic)

### Operation of the safety shield

The safety shield is the large curved part that stands between the furnace and the user during a fusion, to protect against accidental burns when the red-hot platinumware holders slide out at the end of the cycle.

The shield must be manually pulled up to access the platinumware holders. In the fully open position, the shield will rest against a stopper, in the near-vertical position.

Once ready to start another fusion, the shield must be manually pulled down until it rests in lower position.

### **Automatic lock operation**

In normal use when the unit is powered, the safety shield is unlocked when no fusion is ongoing, and will automatically lock when a fusion is started. The shield will unlock by itself at the very end of the fusion cycle, when the cooling fans stop.

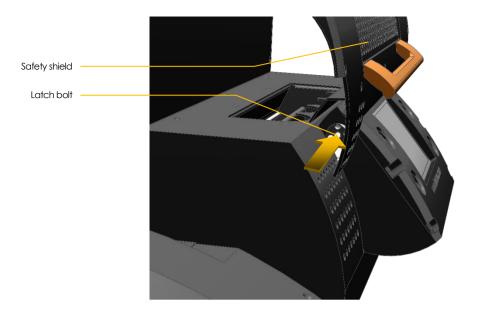
A detector ensures that the shield is in place before allowing the fusion to start.

Katanax recommends working in that default configuration, but it is also possible (at the user's risk) to entirely disable the locking mechanism if preferred. Refer to *Safety shield protection* on page 46 for details.

#### Manual override

When there is no power (e.g. when cleaning or packing the unit), it is possible to override the locking mechanism to manually open the safety shield.

The locking latch bolt can be reached behind the shield, on the left-hand side near its bottom. Use the eraser end of a pencil (or the non-writing end of a pen) to push the latch bolt inwards to the right, and then gently open the safety shield. You can also use your hand to push the latch bolt if you have long slender fingers, but be careful not to pinch the tip of your finger while opening the shield.

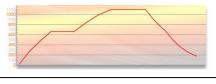


### The main running screen

After the booting screen, you will obtain a display similar to the following (not all buttons/icons will appear, depending on the instruments' settings):



Here is a short explanation of the various zones and buttons:



The graph zone shows the outline of the crucible temperature as a function of time.



The "Start" button, quite obviously, is used to launch the currently selected fusion program.





The crucible temperature icon is depicted with

- the actual measured temperature (in orange), and
- the target temperature, which the crucible heater is in the process of reaching (in gray)





The "ramping" icon informs on the rate at which the current target temperature is to be reached.

When turning the instrument on, this will always show "Fast".



The "beaker loading" icon is used to temporarily make the platinumware holders slide back, in order to easily place the solution beakers in their holes.



The "parameters" button is used to adjust the individual setting of each program step. More details are given at page 33, *Programming the K2 Prime (advanced)*.



This zone is used to select the current program. Click on the arrows to switch to a recipe selection screen.

Clicking on the recipe name itself will allow you to rename it. More details on this are given at page 38, *Managing fusion methods*.

| The Copy button is useful to duplicate an existing program, to create a derived recipe. More details are given at page38, <i>Managing fusion methods</i> .   |
|--|
| The Delete button is used to erase a program from memory. More details are given at page 38, <i>Managing fusion methods</i> .  |
| The Save button is used to write the current program and its parameters into memory. More details are given at page 38, <i>Managing fusion methods</i> .   |
| The Global Parameters button is used to access a screen where general configuration settings can be modified. More details are given at page 44, <i>Global parameters</i> .  |
| The padlock icon/button shows the state of the fusion recipe parameters.  A closed padlock means that the parameters are locked, and a password is required to unlock parameter access. Conversely, an open padlock means that all parameters can now be freely changed.  More details are given at page 33, |
| Programming the K2 Prime (advanced).   |
| Entering the password is also required to modify the parameters of the Global parameters screen.   |

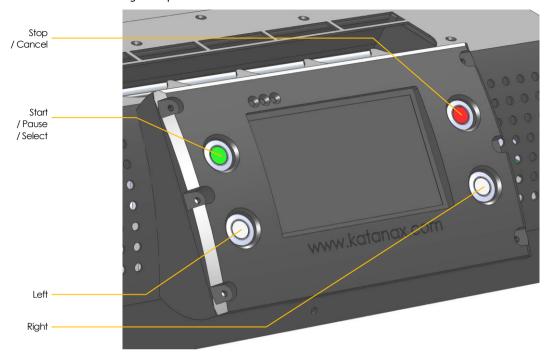
### Electromechanical pushbuttons

In addition to the touch-screen soft-buttons, the instrument is fitted with four (4) regular pushbuttons, located on either side of the screen. These pushbuttons can be used by day-to-day technicians who use the instrument, but do not need to adjust fusion parameters.

The green button (top left) is equivalent to "Start", "Pause/Resume" or "Select", depending on the screen.

The red button (top right) is equivalent to "Stop" or "Cancel".

The last two buttons (bottom left and right) are equivalent to the orange left and right recipe selection arrows.



To start a fusion program, it is therefore just a matter of scrolling through the programs with the right/left arrows (with either the side pushbuttons or touch-screen soft-buttons), and then pressing Start.

### Loading a program

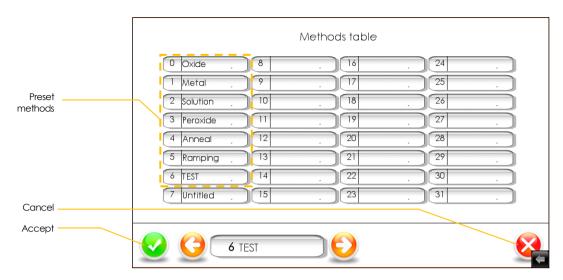
Changing the current program can be made in several ways.

Touching the program number will call the program selection screen.

Touching the left or right arrows on either side of the program name will also call the program selection screen, but will also decrease/increase the program number. This can also be done by pressing the Left or Right mechanical buttons.

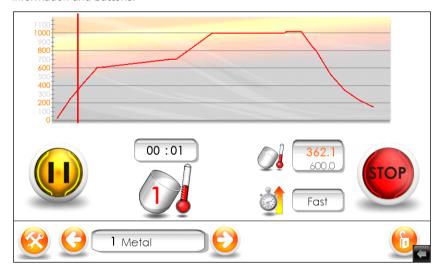
In the program selection screen, you can use the left or right arrows to scroll among the proposed programs, or you can directly select the desired program by touching its name.

Touch the green button to confirm, or the red to cancel.



### **During a fusion**

While the instrument is running, the main screen will display additional information and buttons.



Here is the explanation of the new graphical elements. (Other icons were explained at page 18, in *The main running screen* section.)



The Pause button is used to temporarily "freeze" the ongoing fusion.

In pause mode, timers are suspended, and the current furnace temperature is maintained. Any ongoing motor motion will be continued or completed. This can be useful when some extra time is required to complete an oxidation or dissolution reaction, for example.

Press the Pause button again to resume normal operation.





This cluster represents the elapsed time (mm:ss) since the requested temperature is attained, as well as the currently-running step number (in red).

See page 31, *Description* of the fusion steps, for more information on this topic.



The Stop button, as the name implies, is used to halt an ongoing fusion process, stopping all motors.

This can be used when one realizes that the crucibles or molds are not properly prepared, or in case of emergency, for example.

- Pressing Stop again will cancel the ongoing program and reset the instrument.
- Pressing Start instead will resume the fusion program.

### The right ingredients

From the preceding paragraphs, we already know how to launch a fusion program. There are, however, a few other things than one should know to obtain a perfect disk. These include:

- 1. properly preparing the sample for the fusion,
- 2. selecting the appropriate flux blend from the sample type,
- 3. determining the total mass in the crucible from the mold capacity,
- 4. estimating the flux-to-sample ratio,
- 5. using the appropriate additives, and
- 6. mixing the components together.

#### Sample preparation

Besides the traditional specifications of a sample to be representative, uncontaminated and dry, Katanax recommends that the sample be ground to  $<100\mu m$ . This is to ensure that the fusion be completed within a reasonable time.

Additionally, the sample must be fully oxidized before heating the crucible containing the sample.

**IMPORTANT:** Heating a sample containing metallic species at high temperatures will cause an alloying reaction, damaging the crucible, and possibly damagingthe instrument.

#### Flux blend

Typical fusions use a mix of lithium metaborate (LiM) and lithium tetraborate (LiT). Lithium metaborate alone typically offers a better sample solubility, but generally leads to crystallization of the bead. Tetraborate stabilizes the melt, but limits solubility. Thus, to optimize solubility and obtain perfect glass disks, one must use the correct LiT/LiM flux ratio.

The mixing ratio is determined by the acidity of the sample. Acidic samples require a basic flux (more LiM), while alkaline samples need an acidic flux (more LiT) and neutral samples call for neutral flux (50% LiT + 50% LiM).

Here is a list of common oxides, along with the recommended flux.

| Simple oxide  | Recommended flux          |  |
|---|---------------------------|--|
| K <sub>2</sub> O, Na <sub>2</sub> O, BaO, SrO, Li <sub>2</sub> O, CaO, Ag <sub>2</sub> O, MnO, MgO, PbO, CoO, BeO   | Lithium tetraborate       |  |
| ZnO, CuO, NiO, CrO <sub>3</sub>   | 67% Lithium tetraborate – |  |
|   | 33% lithium metaborate    |  |
| Fe <sub>2</sub> O <sub>3</sub> ,Sb <sub>2</sub> O <sub>3</sub> , ZrO <sub>2</sub> , TiO <sub>2</sub> , SnO <sub>2</sub> , V <sub>2</sub> O <sub>5</sub> , | 50% Lithium tetraborate – |  |
| SeO₃  | 50% lithium metaborate    |  |
| $Al_2O_3$ , $B_2O_3$ , $SiO_2$ , $Bi_2O_3$  | Lithium metaborate        |  |

Naturally, actual samples are generally composed of more than one oxide type, so a rough proportion must be calculated to determine the optimal flux type.

The only exception is when one makes solution. Since making a solution involves the complete dissolution of the melt into an acid, one does not care that the melt crystallizes when it cools; it is not only unavoidable, it is desirable. Therefore, when making a solution, one can pretty much always use lithium metaborate only, to increase sample solubility.

The choice of flux is also governed by the various chemicals that must be added. For example, many samples are initially un-oxidized, and thus need to be oxidized; since borate fluxes only dissolve oxides (metallic material ruins crucibles at high temperatures). Addition of strong acids or bases is often the easiest solution, but flux type must be adjusted accordingly.

### Amount of flux and sample to use

Today's market has seen a proliferation of various mold diameters. Consequently, one must adapt the amount of flux and sample to obtain a full disk that will not overflow out of the mold.

Our recommendation is to measure the actual inner diameter on the bottom of the mold and apply the following formula, to obtain the total mass of sample and flux:

Total mass [g] = 
$$\frac{\text{(Mold diameter [mm])}^2}{150}$$

Thus, for a 32-mm inner diameter mold (recommended diameter), we obtain  $32^2 / 150 = 6.827$  g, which we can round up to 7 g.

This being said, there are also molds on the market that are very shallow (despite the thickness of the metal they are made of). Those molds will require less flux to fill correctly, but leveling of the mold is more critical.

### Flux-to-sample ratio

After the choice of the right flux, the flux-to-sample ratio is probably the second hardest question to answer. This section intends only to explain general concepts. For more specific information, the customer is invited to contact Katanax directly.

To obtain the best readability possible on the analytical instrument, one wishes to put as much sample as possible in the preparation. However, putting too much sample will over-saturate the flux, and leave undissolved sample particles in the disk.

The solubility of samples into the flux being rather hard to predict theoretically, it is recommended to work with the following method:

- 1. Determine the optimal flux type. If unsure, 67% LiT with 33% LiM is a good starting point.
- 2. Using the formula above, calculate the total amount of flux and sample required for your mold size.
- 3. From this mass: weigh 5% sample for 95% flux, directly in the crucible. Mix thoroughly.
- 4. Proceed with fusion, and observe the result.
- 5. If the bead is perfectly homogenous, it is possible to try increasing the amount of sample a little.
- 6. If the bead is milky or dusty (presents tiny particles of undissolved sample), try again with less sample, or change the flux type a little. It is also possible that the sample be not completely oxidized. Just after weighing the sample (before adding the flux), add a small amount of solid oxidizer, liquid acid or liquid base, depending on what reacts better with the sample at hand.

The optimal flux-to-sample ratio is found when all the sample is dissolved, and almost saturates the flux solvent.

Note that increasing fusion temperature does not allow the stable dissolution of more sample. It may quicken the dissolution speed, but when the disk will cool down, a precipitate will appear, or the disk will be prone to spontaneous bursting.

#### The non-wetting agent (NWA)

The non-wetting agent (NWA) acts as a surfactant that makes the melt less prone to sticking to platinumware. Non-wetting agents are halogen compounds (generally containing Iodine, Bromine or Fluorine) and typical formulations include KI, LiI, LiBr, NH<sub>4</sub>I and NaI. Only a few milligrams are required. If in doubt, use about 30 mg of Libr and observe the results.

We strongly recommend using such a non-wetting agent, to lengthen the mold's life expectancy, and to ensure all the melt is transferred into the mold upon pouring.

Katanax also sells flux blends that contain predetermined amounts of non-wetting agent. Please contact Katanax to obtain this time-saving product.

### Oxidizing agents

As previously mentioned, it is of key importance that the sample be oxidized. While it is often safer and easier to oxidize the sample using a liquid acid or base before fusing, it is also possible to use powder reagents to oxidize the sample in a one-step operation.

Typical oxidizers are lithium carbonate ( $\text{Li}_2\text{CO}_3$ , which reacts at around 700-800°C), and lithium nitrate ( $\text{Li}_1\text{NO}_3$ , which reacts at around500-600°C). Several minutes at the reaction temperature must be allowed before heating up further, and temperature ramping can be useful to avoid spills due to too fast a reaction (see page 33, *Programming the K2 Prime (advanced)* for details on ramping). The amount of selected reagent will depend on the sample contents and can be estimated stoichiometrically. An excess of oxidizer is recommended.

#### Manual mixing

Once all the components are selected and weighed into the crucible, some manual mixing is recommended, to improve contact between the various reagents.

In particular, very fine sample particles have been observed to agglomerate, and a manual mixing will help breaking the lumps that might have formed during and after weighing.

Two notable exceptions to this general rule are high-carbonate samples and when using powder oxidizers. In those special cases, one want to first lay some flux on the bottom of the crucible, then add the sample (and oxidizer) on top. Manual mixing would ideally just be done with the sample and oxidizer, because one wants to have the most intimate contact between the sample and the oxidizer. Flux will merely act as a shield at first, protecting the crucible from alloying with the sample. In the case of high carbonate samples, it is best to lay the sample on top of the flux and not mix; the expelled gases will escape more freely.

### Care of the elements

In the K2 Prime, heating elements build a surface oxide layer, which protects them from degradation and lengthens their lifespan. However, this oxide is extremely sensitive to molten flux.

It is very important that no flux ever touch the elements. Element life would dramatically be reduced.

**IMPORTANT**: While it is recommended to remove the crucibles for weighing, some users prefer to leave them in place and transfer the powder from a vial into the crucibles. In all cases, make sure that the crucible external walls are clean before re-installing them in their holder, and make sure that no flux has spilled.

### Care of the platinumware

Crucibles and molds should be considered an integral part of your fusion machine.

As such, care must be taken to ensure that they are free from leftover flux, molten or in powder. If need be, you can use hot 20% HCl (and proper precautions) to clean them overnight.

It is also important that the crucible's and mold's interior surfaces be kept polished, to ensure a smooth melt pour, easy bead removal and good analytical results. A very fine diamond paste with a rotary tool fitted with a soft felt or cotton tip makes for a simple in-house polishing method.

Finally, crucibles and molds are quite fragile and can distort over time. Re-shape these items without delay to restore their original dimensions.

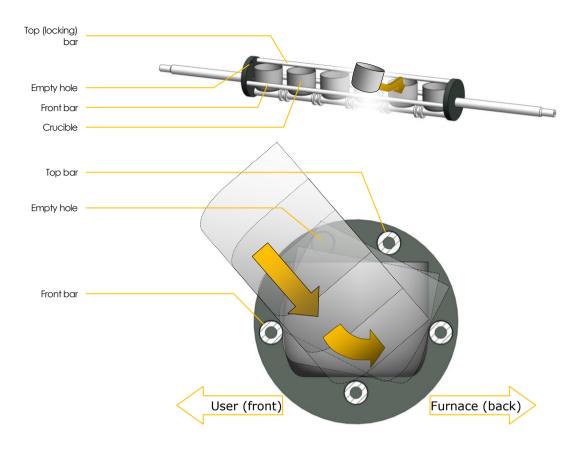
With proper care and fusion method, a crucible a be expected to last for several hundred fusions, while molds typically last longer.

### Crucible installation

Once the crucibles are filled with the proper components, they can be installed in the fluxer holder, one at a time.

#### To install a crucible:

- 1. Simply tilt its top towards the furnace and insert the crucible into its holder, between the top bar and front bar.
- 2. Once the crucible is "inside" the holder, it can be rotated back to its natural straight-up stance.

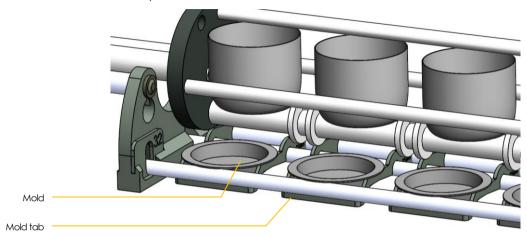


**IMPORTANT**: Make sure that all the ceramic rods / axles are fully inserted into their corresponding holes.

**IMPORTANT**: Katanax recommends that the crucibles be filled when not in the instrument. This allows to avoid accidental powder spills on the holders, and ensures that the crucibles are not installed in the very same position every fusion (in which case they could be prematurely damaged).

### Mold installation

After the crucibles have been installed, one must place a mold in line with each crucible, on the horizontal mold tabs.



**IMPORTANT:** Forgetting to install the molds will cause the crucibles to pour the hot molten glass onto the instrument. In such case, a cavity is designed to receive the hot melt without damage, but sample will be lost.

### A general fusion

Here are the steps required to perform a fusion on the K2 Prime fluxer.

- Turn the instrument on by flipping the rocker switch at the back of the instrument. The main screen appears and the platinumware holders are automatically pulled out of the furnace. Furnace heating is automatically turned on, to reach the stand-by temperature.
- Check crucible and mold holders for possible flux spills from a previous fusion. (See page 54, Flux spillage on holders for details.) If holders look vitreous and dirty, clean the holders immediately; do not start a fusion process with dirty holders, as this could damage the instrument.
- 3. Select the desired fusion program, by clicking on the left and right arrows that flank the current program name.
- 4. In the platinum crucibles, weigh the required amount of flux.
- Add the sample by weighing it directly into the crucibles on top of the flux. Mix if no solid oxidizer is to be used and the sample is low in carbonates.
- 6. Prepare solid oxidation, if required.
  - 6.1 Add a suitable solid oxidizer (generally, a nitrate or carbonate) in the crucibles.

- 6.2 Mix thoroughly with the sample. (Try to leave the bottom layer of flux untouched.)
- 7. Add the non-wetting agent if it is not already integrated within the flux. Solid non-wetting agent should be thoroughly mixed with the flux. Making an aqueous solution with the solid salts can also prove very convenient, and can be pipetted on top of the dry ingredients.
- 8. Place crucibles in the holders. **Important:** make sure that they are properly installed. See page 27, *Crucible installation*, for details.
- 9. Place molds on their holder. **Important:** do not forget to install a mold under each crucible. See page 29, *Mold installation*, for details.
- 10. Touch Start to launch the fusion. If the preset temperature is not yet reached in the furnace, a few minutes' delay will allow sufficient heating, then the holders will automatically enter the furnace. The door opens and closes automatically.
- 11. Upon heating, the sample reacts if an oxidizer is present. Then, the flux melts and dissolves the sample. The mold is heated at the same time. At the end, the door opens to let the crucibles out, and those are tilted to pour into the molds. A blower cools the disks.
- 12. At the end of the cycle (when the cooling blower turns off), carefully pick up the glass disks. **Important:** They may still be very hot, depending on the mold weight and program parameters.

### Making solutions

**IMPORTANT:** Before attempting to make solutions, it is important to remove the middle section of the mold holder, which can otherwise interfere with the top of the beakers.

**IMPORTANT:** Katanax does not recommend attempting to prepare solutions in a fluxer not fitted with the optional solution magnetic stirrers.

When making a solution, the process is quite similar, but the mold installation changes for the following:

- 9. Place beakers in instrument.
  - 9.1 Fill PTFE beakers with about 100 ml suitable dilute acid. (Nitric acid is the most commonly used.)
  - 9.2 Add magnetic stirring bars.
  - 9.3 Press the "load beaker" button to slide the platinumware holders back, against the furnace door.
  - 9.4 Put the beakers on the agitator, in their respective holes.
  - 9.5 Optionally, press the "load beaker" button again, to move the platinumware holders to the front and clear the door opening.

The magnetic agitation system is always active.

A parameter in the pouring step can be toggled to indicate a "solution mode". This will automatically slide the holders in "load beaker" position immediately after pouring, thus keeping the holders away from the vapors produced by the hot acid.

The fluxer beeps when the beakers are ready to be picked up.

### Description of the fusion steps

All fusion programs in the K2 Prime are built the same way, and have seven (7) steps. Here is the list of those steps, along with the corresponding icon:



### Heating 1

Typically used to pre-heat the sample, with little or no agitation.



### **Heating 2**

Typically used to oxidize the sample, with little or no agitation.



#### Heating 3

Typically used to melt the flux.



#### **Heating 4**

Typically used to dissolve the sample in the flux.



#### **Pouring**

Used to transfer the crucible contents into a mold or beaker.

Not used for some preparations, with peroxide fluxes for example.



#### Cooling 1

Used for natural-cooling of the mold, or stirring of the solution



#### Cooling 2

Used for blower-cooling of the mold

Each step is launched when the preceding one is completed. It is also possible for a step to have a null duration (i.e. zero seconds), and would simply be jumped over. Most fusion programs will not use all heating steps.

Note that all heating steps  $(1\ \text{to}\ 4)$  are identically structured and could be used interchangeably.

Also, some of the steps have built-in on/off switches that allow extra actions to be executed, or sometimes to turn off the fusion step itself.

Manual edition of step parameters is the subject of the next chapter.

# Programming the K2 Prime (advanced)

When specific sample types do not seem to be easily processed by a preset fusion method, it is necessary to manually modify the parameters of critical fusion steps.

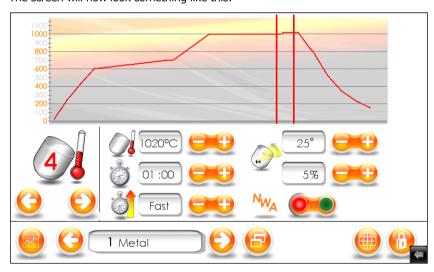
Viewing the fusion parameters

Without risking to change a parameter, any user can check the current values and settings of the current program, step-by-step. This is done by pressing the "Settings" icon, in the lower left corner of the main running screen.

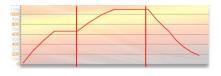


The Settings icon allows to toggle to a screen where the parameters for a given fusion step are displayed. Nothing can be changed unless the advanced mode is unlocked (see page 37, *Unlocking the advanced mode*).

The screen will now look something like this:



Let us now understand the meaning of each symbol.



We are now familiar with the graph zone, which shows the outline of the crucible temperature as a function of time.

The fusion step being edited is represented as the portion of curve between the two vertical red lines.



The large crucible with red thermometer represents a heating step, and the numeral "4" indicates that we are now viewing the parameters of step "Heating 4".

The Left and Right arrows are used to scroll among the steps of the current program.



The small crucible with red thermometer icon is placed just besides a cell where the target crucible temperature is displayed. (The Minus and Plus buttons will be used to change the parameter.)



The stopwatch icon is placed just besides a cell where the step duration is displayed. Note that step durations are calculated *after* the required temperature is reached.



The stopwatch with arrow icon is placed just besides a cell that indicates how the target temperature will be reached.

Most fusion applications can use the "Fast" setting, but oxidation steps often call for a slow heat-up rate. This is called "ramping".



The crucible with angle symbol is used to refer to two rocking parameters.

The top one is the amplitude of the rocking.

The bottom one is the rocking speed.



The  $^{\circ}O_2''$  icon (not shown above) refers to the ventilation of the furnace, achieved through the activation of an optional custom-made oxygen injector, or by repeatedly opening the furnace door (default configuration).

Available for steps 1, 2 and 3 only.



The "NWA" icon refers to the activation of a step that allows a manual addition of non-wetting agent.

Available for step 4 only.



The icon in the lower left corner has now switched to a graph icon.

Pressing this button will bring you back to the main running screen.

If we scrolled fusion steps towards the right, until we reach the pouring step, we would obtain a screen similar to the following:



Icons and symbols have the following meaning:



The large crucible pouring into a mold shows that we are now viewing the Pouring step parameters.



This block of icons shows that the pouring is On (thus the "green light").

The parameters to the top right show the crucible angle upon pouring (in degrees), as well as the pouring motion speed (in percent).

The bottom line shows the current parameters for the final shaking of the crucible after pouring.



This block of icons shows that the annealing is activated (thus the "green light").

Annealing is very rarely used, but in the most specific applications. It pulls the full molds *back* into the furnace at the given temperature, *after* pouring, for the given duration.

Note that annealing and de-gassing (see below) cannot be used in the same program.



This block of icons shows that the degassing is activated (thus the "green light").

De-gassing is very rarely used, but in the most specific applications. It allows to *decrease* the temperature for the given duration, right *before* pouring.

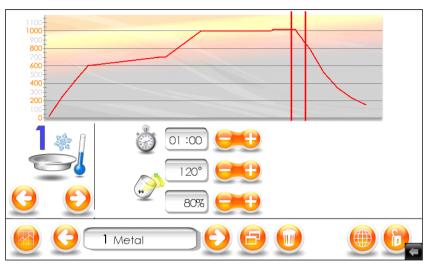
Note that de-gassing and annealing (see above) cannot be used in the same program.



This icon switch provides the user with the possibility to configure a fusion method as a "solution-making" method, and when turned on, will automatically position the holders in optimal location for beaker loading/unloading.



This block represents the settings of the "shaking" feature. After the pouring action, one can program the crucible to shake up and down for a number of times at a given amplitude. Again, scrolling to the next step will show us the available parameters for a cooling step.



And we now recognize the duration parameter, as well as the crucible tilting angle and motion speed. The two crucible-related parameters are used to control the straightening up of the crucible holder. There is typically no rocking motion available in this step, except if the pouring was previously turned off; the system would then assume that the user wishes to perform a "non-pouring" fusion (e.g. pyrosulfate and peroxide).

Typically, the tilting angle of "Cooling 1" will be the same as the pouring, and the tilting angle of "Cooling 2" will be ninety degrees (90°) to prevent residual drops from sliding on the outside wall of the crucible, or fall onto the cooling bead. This will give some time for the flux to pour completely out of the mold during "Cooling 1", while partially straightening the crucibles when the blower starts at the beginning of "Cooling 2".

# Unlocking the advanced mode



Before being allowed to manage fusion program and edit parameters, one must enter the correct password. To do so, click on the padlock icon/button.



After touching the padlock button, a numeric keypad will pop up, ready for password entry.

Type the password, which is 2014.

If you make a mistake while typing, press CR ("clear").

If you summoned the numeric keypad by error, you can close it by pressing ES ("escape").

Once the password is correctly typed, press ENT ("enter") to confirm. The numeric keypad will close, and the padlock icon will now be displayed as unlocked.



This icon informs you that you can now modify the fusion program parameters, but also manage the fusion methods (i.e. copy, delete and save). You are now in "advanced mode".

**NOTE:** It is not possible to modify the parameters in the preset programs, and so even entering the correct password will not "unlock" the padlock icon. However, the instrument remains in "advanced mode", and switching to a custom fusion program will unlock the padlock and allow parameter edition.

To close the advanced mode (i.e. "re-lock" the padlock), simply click the icon and enter an incorrect password on the numeric keypad.

## Managing fusion methods

Fusion methods can be managed just like files on a computer. In the main screen, you can press the icon corresponding to Copy, Delete and Save. Note, however, that the "advanced mode" but first be accessed in order to perform any of the following actions.

#### Copying



The Copy button is useful to duplicate an existing program, to create a derived recipe. Hence, begin with a preset program that is close to the sample type you want to process, and then you will be able to fine-tune the parameters to suite your specific sample. After clicking on

the icon, a window will ask for a confirmation. Click "Yes" to proceed, or "No" to cancel.

#### Renaming

Once a method is copied, you will be automatically brought into that copied program, named "Untitled". We suggest that you immediately rename this with some name that is relevant to your application. To rename the program, click on its name (in this case, "Untitled"), and a full keyboard will pop up. (Note that renaming a preset program is not allowed.)

#### **Deleting**



The Delete button is used to erase a program from memory. Once a program is erased, it frees the corresponding memory slot, and it cannot be recovered. Furthermore, preset programs cannot be deleted.

#### Savina



The Save button is used to write the current program and its

parameters into memory. This icon will appear automatically when the user changes a parameter value or a setting in a program. Otherwise, the icon is not shown.

# Preparing a fusion program

To build your first fusion program, you must first select a preset program template that will be used as a starting point to design your own program. In most cases, the Oxide program is a good all-around program. Copy it under your desired name, as described above.

Once this "editable" program exists, you can adjust parameters to suit your sample.

#### Heating steps

Heating steps all have the same structure. Hence, if you need only two temperature plateaus, you could use Heating 1 and Heating 2, or Heating 1 and Heating 3, and so on, without affecting anything. For standardization purposes, Katanax tends to use the last heating steps, and leave the first ones empty when not needed.

#### **Temperature**



Furnace temperature can be adjusted by pressing on the plus and minus buttons besides the crucible temperature icon.

Note that he K2 Prime firmware prevents the user to create a decreasing temperature. The instrument will prevent a heating step from having a temperature that is lower than an earlier step. Also, the instrument will automatically increase temperatures of subsequent steps to match the step being edited.

#### Examples:

Suppose that we have a program with Heating 2 at 500°C and Heating 3 at 700°C. You will not be allowed to decrease Heating 3 below the temperature of Heating 2, i.e. 500°C.

Now suppose that the program has Heating 1 at 900°C and Heating 2 also at 900°C. If you increase temperature of Heating 1, the temperature of Heating 2 will be automatically increased by the same amount.

User is advised that too high temperatures will affect the life expectancy of the heating elements, but can also lead to analytical problems, due to evaporation of the flux.

Katanax does not recommend exceeding 1050°C when using lithium borates.

Katanax does not recommend exceeding 1000°C when using sodium borates.

Heating above those temperatures could cause flux evaporation that could bias the subsequent analysis. Please contact Katanax if you feel that your sample type needs higher temperatures.

#### **Duration**



Step duration (mm:ss) is also adjusted by pressing on the plus and minus buttons. The actual step timer will start once the furnace

has reached the temperature set for this step. Hence, the length of a step is actually the sum of the time required by the furnace to increase up to the step temperature, plus the duration parameter. For each step, the duration parameter is limited to 19 minutes and 55 seconds, except if ramping is in use (see below).

#### Ramping



The ramping parameter determines how fast the furnace will increase its temperature to reach the one set in the current step. In most application, we want the furnace to heat up as

fast as possible, but it is also possible to set this parameter (by pressing on the plus and minus buttons) to limit the heat-up rate. The other ramping values (besides Fast) are given in °C/minute.

Slow heat-up rates are particularly useful with a solid oxidizer, when we want it to react slowly over a temperature range of about 100°C.

#### Crucible rocking speed and amplitude



The crucible content is mixed by a back-andforth rocking motion, whose amplitude (in degrees) and speed (in % of the max) can be controlled by means of the plus and minus buttons.

Typically, initial heating steps call for very little rocking. This allows for the oxidizer to react, and for flux to melt without risking overflowing from the crucible (molten flux takes up less volume than powder flux).

When pouring approaches, speed and amplitude can be used more generously. One exception would be with samples containing gases. Those samples cause bubbles in the melt and de-gassing the melt is sometimes better achieved with very slow speeds before pouring.

#### Oxygen injection (available in steps 1-3 only)





When the instrument is fitted with the custom-made oxygen injector manifold, setting this parameter to "on" will open the solenoid valve for the duration of the step, thus allowing the oxygen to flow inside the

furnace and help oxidize the sample.

However, by default, this parameter is used to ventilate the furnace cavity by briefly opening the door every 30 seconds. This is recommended when using halogen non-wetting agents, which can otherwise damage the instrument.

#### Non-wetting agent (available in step 4 only)





When this parameter is set to ON, once the furnace reaches the temperature set for Step 4, the door will

automatically open and the platinumware holders will be slid outside. The user then manually adds the desired amount of non-wetting agent directly into the hot crucibles, and then presses Start to resume the normal fusion.

While this feature can be useful with extremely sticky samples, one must keep in mind that the quantity of non-wetting agent injected manually may not be exactly the same for each crucible, and thus could lead to analytical issues.

#### Pouring step

The pouring step is when the crucible is tilted forward quickly, to empty its contents either into a mold or a beaker containing acid. However, pouring can be turned off altogether, for those fusion types where pouring is not desired: fusions in sodium peroxide or potassium pyrosulfate.

#### Basic pouring parameters



Pouring can be completely turned off and on by pressing the red and green vertical switch.

If pouring is on, the crucible tilting angle and speed can be controlled with the plus and minus buttons.

Generally, a pouring angle of 120° with a speed of more than 30% works

well. Adjustment is sometimes required to adapt to melt viscosity and mold size.

#### **Annealing**



Annealing is very rarely used, except in very specific applications, with highly unstable glass disks (i.e. disks that always crack or crystallize). When activated (by means of the horizontal red and green switch), the instrument will pull the full molds (after the pouring) back into the furnace, at the given temperature, for the given duration (adjustable with the plus and minus buttons).

Generally, however, cracking and crystallizing disk problems can be cured by proper control of the cooling (see below).

#### Crucible shaking



In some cases, a droplet will remain stuck inside the crucible. Once cooled, it can easily be pinged off. However, in some instances, one

wishes to completely transfer the melt out of the crucible. This is mostly done with the help of non-wetting agent, but can also be helped with a mechanical shaking of the crucible after pouring.

The shaking feature is activated by setting the number of shaking motions (1x to 25x) and the shaking amplitude (1° to  $20^{\circ}$ ). Press the right half of the button to increase the parameter, or the left half to decrease. Setting those parameters to zero will cause no shaking.

The cooling process is normally divided into two distinct steps. The first cooling stage (Cooling 1) is a natural-convention cooling, that is, without any forced-air cooling. This allows for the melt to completely fill the molds while the molds are as hot as possible, and is of key importance in the stabilizing of the melt.

Indeed, in most cases, if we were to start the forced air circulation immediately after pouring, the bead would not cool down uniformly, and residual thermal stresses would remain in the solid disk. This can cause hazardous bursting of the produced disk, in the next minutes, hours or even days.

On the other hand, and again there are exceptions, if the blowers were to start too late (Cooling 2), a crystallization reaction could occur if the molecules have enough time to arrange in an orderly fashion. Crystallization reaction cause the transparent melt to become milky-opaque, typically from the edges towards the center of the mold, pushing the still-liquid melt to the center and upwards as crystallization progresses, thus creating a volcano-like structure. Therefore the solution resides in finding the correct time for still-air cooling (Cooling 1), and then the blower can start and work until the molds are comfortably cool to the touch. Hence, the duration parameter is very important, especially for "Cooling 1".

#### Duration



Step duration (mm:ss) is adjusted by pressing on the plus and minus buttons. Generally speaking, a "Cooling 1" duration of

one to two minutes is a very good starting point. Thereafter, "Cooling 2" can be set for as many minutes as needed, and this parameter will be roughly proportional with the melt and mold combined weight.

#### Crucible position after pouring



In a typical cooling step, the two cruciblerelated parameters are not used to control the crucible rocking motion, but rather to control the straightening up of the crucible holder, after the pouring.

Typically, the tilting angle of "Cooling 1" will be the same as the pouring (typically  $120^{\circ}$ ), and the tilting angle of "Cooling 2" will be ninety degrees (90°) to prevent residual drops from sliding on the outside wall of the crucible, or fall onto the cooling bead. This will give some time for the flux to pour completely out of the mold during "Cooling 1", while partially straightening the crucibles when the blower starts at the beginning of "Cooling 2".

Note that, if pouring was turned off, then the fluxer is in "non-pouring" mode, and so will allow for a rocking at this step, just like a normal heating step. This is useful to spread the melt onto the crucible walls when making peroxide or pyrosulfate fusions.

# On-the-fly editing

During a fusion, it is also possible to edit parameters on the fly, that is, while the fusion program is running.

To do so, simply edit parameters as explained in the previous paragraphs (from page 33 onwards).

Note, however, that there are logical limitations, and the firmware will automatically limit the accepted parameter range to prevent nonsensical or error-causing combinations. Some parameters may also be accepted, but take only effect on the next fusion cycle.

# Global parameters

In addition to recipe-specific parameters, your fluxer provides extra versatility through flexible parameters that will apply to all fusion programs.



To modify the global parameters, first unlock the advanced mode (see page 37, *Unlocking the advanced mode*), then touch the Global Parameters icon that is now available on the main display screen.

#### Language

In the global parameters page, you can change the instrument's interface language by selecting your preferred language in the list.

#### Holding temperature offset

In order to keep the furnace up and ready for the next fusion, the instrument can maintain power to the furnace to keep it hot. This will help skip the initial ramping time, thus quickening fusions and increasing sample throughput.

In the global parameters screen, you will find a slider for "Holding temperature offset". Indeed, the holding temperature will be calculated *based on theStep 1 temperature*, plus or minus the global parameter offset.

#### Example:

In your fusion program, you have set Step 1 with a temperature of 700°C, and the global parameter for holding temperature offset is +50°C. Then, no matter how the parameters for the rest of the program are set, once the fusion is complete, the furnace will maintain a temperature of 700 + 50 = 750°C.

The offset can be positive or negative.

Using a positive offset, say +200°C, will prepare the furnace to receive the room-tempered platinumware holders at the beginning of the process. Indeed, a temperature drop in that order of magnitude is typically observed when the door opens and the linear motion system slides the platinumware holders into the furnace.

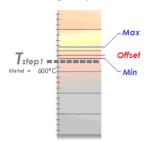
On the other hand, using a negative offset will save energy and increase furnace life.

Keep in mind that the holding temperature depends on the fusion program currently loaded on the interface screen. Changing the fusion program could therefore change the holding temperature (because all fusion programs do not necessarily have the same temperature set at Step 1).

When the instrument is turned on, the furnace will automatically be activated to reach the holding temperature.

#### Startup tolerance

Basically, when you start a fusion, the crucibles will enter the instrument only once the furnace has reached the calculated holding temperature (based on the temperature of Step 1 and the selected offset value). Therefore, there may be a delay when you press Start, to allow for the furnace to heat up or cool down to the holding temperature.



The user can, however, set a range of permissible temperatures, in which the crucibles are allowed to slide in the furnace. This is called the startup tolerance.

Using two sliders, you can configure the blue upper and lower limits (relative to the red main offset) between which you will permit the holders to enter the furnace. You can set independently the upper limit (called *Max*: [0 to +200]) and lower limit (called *Min*: [-200 to 0]), and the platinumware holders will

be allowed to enter the furnace at the beginning of the fusion, only when the furnace temperature  $T_{\text{furnace}}$  is:

$$T_{step1}$$
 + Offset + Min  $\leq T_{furnace} \leq T_{step1}$  + Offset + Max

(where  $Min \leq 0 \leq Max$ )

Ideally, leaving both min and max to zero (0) will ensure the best possible repeatability conditions.

However, in special cases to enhance productivity, one may want to allow a wider temperature range. Examples of such situations include when a particular fusion method starts with a low-temperature oxidation step and ends with a high-temperature pouring step.

#### **End beep**

When a fusion cycle is completed, the instrument will emit a series of beep sounds. By default, the fluxer will beep for 5 seconds, but this period can be extended up to one hour in 10-minute increments.

# Automatic shut-off delay

When the instrument is idle for a certain period of time, the heating will be automatically turned off, to save energy.

This delay can be changed by the user, depending on the context of use of the instrument. Enter the global parameters screen, and slide the "Automatic shut-off delay" slider to the value of your choice.

# Safety shield protection

By default, the checking of the safety shield position and locking should be enabled, to maximize protection. However, it is possible to disable this security feature.

**WARNING**: Disabling the safety shield protection can lead to serious injuries by extreme heat. User is advised that doing so it at the user's sole risk.

#### **Fusion counter**

In the global parameters screen, there is a read-only parameter that displays the number of fusions since the instrument was built, not unlike the odometer on a car.

# Special parameters

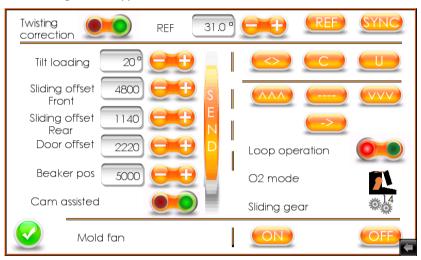
This section presents an advanced interface window that can be used to adjust the offset sensitivities of various electro-mechanical sensors on the fluxer.

WARNING: Changing these parameters should only be done by trained personnel. Incorrectly setting these parameters could cause damage to the instrument.

# Accessing the Special parameters

To invoke the Special parameters window, Press on the padlock icon at bottom right of the main screen and enter the following code "2206" followed by ENT key.

The following window appears:



# Twisting correction

The top section enables (default) or disables, and adjusts the automatic twisting correction. At the end of each fusion, the crucible holder automatically rocks backwards to an angle larger than those allowed in fusion parameters. This movement causes each side of the holder to bump against a mechanical stopper, while the stepper motors slip magnetically. This readjusts both sides of the holder in the same plane, therefore effectively cancelling any twisting of the holder.

Pressing on the ``-/+" button will be used to set an angular position, to be set where a rotation limiter touch lightly. This will be useful when installing new crucible holder shaft couplings.

The "REF" button rotates the holder to the offset value set in the REF field. That is how the user can confirm whether the offset is right or can be further fine-tuned.

The "SYNC" button starts the actual anti-twisting motion, so that one can ascertain that the action is done correctly. With the correct adjustments, three distinct knock sounds should be heard.

**NOTE**: The complete adjustment procedure is described at page 74, *Angular adjustment of the crucible holder shaft couplings.* 

#### Offsets

The middle left section is used to adjust the various mechanical motions.

**IMPORTANT**: None of the parameters below is actually memorized until the user presses "SEND".

#### Tilt loading

This parameter controls the tilting of the crucibles holder to facilitate the manual loading and unloading of the crucibles.

- 1. Enter the desired value using the "- / +" button. (Katanax recommends setting for this parameter at 20°.)
- Press "SEND" (vertical button) to transfer the parameter to the motor controller.
- 3. Press "U" to order the fluxer to move the crucible holders in loading position, at your set angle.
- 4. Test if this angle is convenient by inserting a crucible in the holder, and fine-tune the parameter if needed.

#### **Rocking offset**

This is used to fine-tune the verticality of the "reset" position (i.e. when the crucibles' openings are all upwards, ready for filling).

Increasing the parameter will cause the crucibles to reset to a position where their openings point away from the user.

Oppositely, decreasing the value will cause the crucibles to reset towards the user.

When used in conjunction with the twisting correction, this parameter should not be outside the (-40 to +40) range. If other values need to be used, then a mechanical adjustment of the crucible holder is required.

#### Sliding front offset

This offset controls the position at which the holders slide outside the furnace, to the front. Typical value is around 5000.

Increasing the value will cause the holders to stop later, closer to the user.

Decreasing the value will cause the holders to stop earlier, away from the user.

40 steps are approximately equal to 1 cm.

#### Sliding rear offset

This offset controls the position at which the holders slide inside the furnace.

Increasing the value will cause the holders to stop later, away from the user (i.e. deeper in the furnace cavity). Typical value is around 150.

Decreasing the value will cause the holders to stop earlier, closer to the user.

40 steps are approximately equal to 1 cm.

#### Door offset

This offset controls the position at which the door motors stop the closing motion. Typical value is around 1000.

Increasing this parameter forces the door to try to close further down.

#### Beaker pos(ition)

This parameter controls how far back the holder will slide when the user presses on the "beaker loading" icon.



Increasing the parameter will cause the holder to move closer to the furnace, to allow for easy beaker loading.

#### Cam assisted

This parameter controls how the door motors are powered, and must be set strictly according to the installed hardware on your machine.

The parameter must be set to "ON" (green light) on recent units, where a cam/lever system is fitted in the back of the furnace.

The parameter must be set to "OFF" (red light) on older units, which had 4 springs and no lever.

(When the parameter is set to "ON", power to the motors will automatically be removed when the door closes.)

# Movement testing

To check that the parameters entered and sent work correctly, it is possible to start motions independently.

#### The <> button

This button starts the rocking motion, using the parameters (speed and amplitude) of the step 4 of the current program.

#### The C button

This button starts the pouring motion (i.e. it rotates the crucibles forward to the pouring angle).

#### The U button

This button resets the position of the crucible, to the vertical (loading angle).

#### The ^^^ button

This button will cause the furnace door to open and the holders to slide inside.

#### The ---- button

This button "pauses" any ongoing motion.

#### The vvv button

This button will cause the furnace door to open and the holders to slide outside.

#### The -> button

This button resumes the motion previously paused by the "----" button.

#### The "Loop operation" button

Once armed to "ON" (green light), this setting will cause the next started fusion program to be run in endless loops. Right after the last cooling step, the holders will re-enter the furnace and the fusion program will start over.

Pressing the Stop button cancels the Loop operation, and the instrument resumes normal operation. (The "Loop operation" switch then automatically toggles back to "OFF".)

#### O<sub>2</sub> mode

This parameter was originally used to control only the oxygen injection in fusion steps 1-3, by means of a custom injector.

When such a hardware is not installed (in most cases), the same name also refers to the ability of the furnace to open and close its door repetitively. This is very useful to vent out any corrosive gases produced by the reaction, in particular halogens from the non-wetting agent.

Click on the icon to switch between the two modes:



Door opening-closing to vent out halogens (default).



Oxygen injection toggle (requires special hardware).

#### Sliding gear

This setting should not be changed. (It corresponds to the number of teeth on the linear motion system driving pulley.)

#### Mold fan (on / off)

These buttons start and stop the cooling fan, for testing purposes. This does not change the way the fan works in normal fusion programs.

# Fusion troubleshooting

This section presents the most common fusion-related problems. For specific assistance, please do not hesitate to contact us (see page 91, *Contacting Katanax*).

#### Disk cracks

Disk cracking occurs when there are internal stresses within the glass bead. The specific causes can vary as follows:

#### Glass disk sticks to the mold

If disk top surface is concave (disk adheres to the mold walls), add non-wetting agent at the beginning or during the fusion. Katanax recommends lithium bromide.

#### Glass disk contains undissolved particles

Some sample may not be completely dissolved. Make sure that sample is fully oxidized or decrease amount of sample.

#### Glass disk is improperly cooled

Generally, cooling a disk too fast can cause its cracking. Allow more time for the still-air cooling (i.e. before starting the cooling blower).

#### Disk crystallizes

Crystallization reaction cause the transparent melt in the mold to become milkyopaque during cooling, typically from the edges towards the center, pushing the still-liquid melt to the center and upwards as crystallization progresses, thus creating a volcano-like structure.

#### Inappropriate flux

Crystallization will occur if too alkaline a flux is used (i.e. too much lithium metaborate). Increase the proportion of tetraborate to compensate.

#### Improper cooling

Natural convection (still air) cooling is too long or too short. Edit convection cooling duration parameter manually.

Generally, crystallization occurs when the natural convention cooling is too long.

#### **External contamination**

In some circumstances, dirt, dust or other small debris can be sucked into the blower and land onto the glass bead. This type of crystallization will develop from

this single point on the surface and radiate outwards. Cleaning the instrument's base and surroundings will correct the problem.

### Incomplete disk

This symptom causes disks that have the shape of a moon crescent, i.e. a circle with a missing section.

It is caused by the mold not containing enough flux, or by the mold not being leveled. Level instrument/mold holder or add flux accordingly.

#### Nonhomogenous disk

When holding the glass disk in front of a light source, if you can observe powdery residues, then some sample may not have dissolved.

#### Sample is not fully oxidized

As we know, un-oxidized sample cannot be dissolved in flux, and will also cause damages to platinumware. Make sure to use the proper type and amount of oxidizer.

#### Oversaturation

A fusion being a dissolution reaction, it is perfectly possible to obtain an oversaturated bead. Simply reducing the amount of sample will correct the problem.

#### Improper arindina

If the sample is too coarse, or if it lumps together easily, then the time allotted by the fusion program might not be long enough.

One can either extend the fusion duration, or grind the sample to a finer granulometry. We recommend smaller than 100  $\mu$ m. Also, a manual mixing of the sample with the flux can often prevent the lumping issues.

Sometimes, the small "dust" is actually gas (see below).

#### **Bubbles in disk**

Typical with carbonate samples, this phenomenon exhibits gaseous bubbles that remain imprisoned within the glass disk.

In many cases, simply placing the sample on top of the flux and *not* mixing will allow the sample to de-gas and thus avoid this problem. However, some samples are known to make lumps and become harder to dissolve if not mixed; if that is the case, then simply allow a period of slow mixing for an extra minute or two, just before pouring.

# Periodic inspection

This instrument requires some regular checking, which is very important to keep your instrument up and running.

Katanax knows that a broken instrument in a laboratory setting causes sample back-up and unnecessary costs. That is why this manual comprises not only a Periodic inspection section, but also a Service operations chapter (see page 60), which guides the user in a step-by-step fashion through operations that sometimes need to be performed on-site.

If unsure, do not hesitate to get in touch with a Katanax technician (see page 91, Contacting Katanax).

Note that no modifications of the instrument, except those explicitly described and permitted in this manual, are allowed. Any undue modification automatically cancels the warranty and could endanger the user's life.

#### Warning

**IMPORTANT**: Some of the procedures described in the following pages imply a risk of death by electrocution; those procedures shall be executed only by trained personnel.

## Inspection schedule table

| Frequency                     | Checkpoint                              | Description   | Action<br>(if problem<br>found)                | Page |
|-------------------------------|---|---|--|------|
| Every<br>fusion               | Flux spillage on holders                | Check for flux deposits   | Cleaning                                       | 54   |
| 1 month <i>or</i> 300 cycles  | Holders alignment<br>and functionality  | Check for broken ceramic parts, bent metal parts  | Adjust or replace damaged parts                | 55   |
|                               |   | Check alignment, level and position   | Adjust as needed                               | 68   |
|                               | Visual integrity of<br>heating elements | Check elements integrity, section and pitch regularity                                      | Replace  | 56   |
| 3 months<br>or<br>1000 cycles | Element terminal connections            | Check for correct tightening and oxidation  | Re-tighten or<br>replace                       | 56   |
|                               | Door springs tension                    | Check spring strength and oxidation   | Shorten or replace                             | 57   |
|                               | Furnace cleanliness                     | Check that furnace is clean and not cracked   | Replace or clean                               | 57   |
|                               | Thermocouple junction                   | Check junction  | Replace  | 58   |
|                               | Door safety switch                      | Check for corrosion or<br>blackened contacts  | Replace  | 58   |
|                               | Linear motion system                    | Check proper working, clean,<br>lube carriage (not rail), belt<br>tension, pulley alignment | Adjust, lubricate carriage (not rail) or clean | 59   |

### Flux spillage on holders

This procedure describes an easy, yet crucial, step to be completed before each fusion cycle. Flux spillages (residues), if left un-cleaned, can lead to a rapid deterioration of key components of the fluxer.

- 1. This visual inspection should be done every fusion cycle.
  - 1.1 Look for vitrified, darker or colored spots on the ceramics or metal brackets that make up the holders.
  - 1.2 In the case that a spillage occurs on one or more parts of the holders, clean or replace immediately. Metallic and white ceramic parts of the holders are slowly dissolved by the flux when it is molten, but will quickly degrade when cooling, as the ceramic parts could break.
  - 1.3 See holders section of this manual to remove/replace/disassemble the holders.
- 2. If parts are found to be contaminated with flux deposits, carefully disassemble the parts in question and apply one of the following methods.
- 3. For cleaning ceramic parts

**WARNING:** Do not use these procedures to clean platinum crucibles or molds: it will cause irreversible damage to precious metals.

- 3.1 In a large container (e.g.: 2 liter Pyrex beaker), mix together a 20% Ammonium nitrate (NH4NO3) solution with a 20% Hydrochloric acid (HCl) solution.
- 3.2 Put your container on a hot plate and keep the solution warm at 80°C. (The cleaning solution will not work well until 80°C is reached.)
- 3.3 If your hot plate is equipped with a magnetic stirrer, you can add a magnetic bar in the beaker to agitate the warm solution and accelerate flux dissolution.
- 3.4 For a small or very recent spillage, 30 minutes to 1 hour should be enough to clean it.
  - For a larger or older spillage, up to two hours could be required.
- 3.5 In case of large spillages, a diamond disc mounted on high-speed rotary tools (e.g. Dremel™) can be used to remove major parts of spillage and then the solution can be used to finalize the cleaning.
  - If a rotary tool is used, care must be taken in order not to grind the ceramic part itself.
- 4. For cleaning metal parts (not platinumware)

It is possible to separate metal parts of the holders (but not platinum items) from ceramic parts using the methods described above for cleaning the ceramics.

However, such methods should not be applied for too long; metal parts should be removed from cleaning solution immediately when separated from ceramics.

**Note:** These methods will remove the protective oxide layer, and so re-cooking the bare metal parts at 1000°C for an hour in a clean oven is highly recommended.

# Holders alignment and functionality

This procedure describes how to ensure that both the crucible and mold holders are properly positioned, and in working condition.

- 1. Procedure preparation
  - 1.1 Let the instrument cool down, and turn it off.
  - 1.2 Unplug it from the power outlet.
- 2. Inspection of the holders
  - 2.1 Verify that holders are perfectly horizontal.
  - 2.2 Verify that flux spillage is not present on holders anywhere. (Refer to appropriate section in case flux deposits are found.)
  - 2.3 Verify that crucibles holder is free to rock and pour into the molds, without rubbing or hitting anything. To do so, manually move the crucibles holder to simulate the movement of rocking and pouring.
  - 2.4 Verify that the crucible holder ceramic tubes, those crossing from left to right BlackDisk end-plates remain very gently compressed, so that the ceramic tubes cannot move freely sideways.
  - 2.5 Using the appropriate tools, verify that screws that are used to secure the cantilever ceramic rods supporting the mold holders are well tightened.
  - 2.6 Manually open the door and gently push the holders inside the furnace. Then, verify that mold holders have enough free space to move inside the furnace slots. A minimum clearance of 1 mm should be observed in all directions.
  - 2.7 Keeping the door open and the holders inside the furnace, verify that the crucible holder is free to rotate inside the two furnace slots. Verify that when crucible holder is in horizontal position, it can move freely through the slots during sliding operation. A minimum clearance of 1 mm should be observed in all directions.
  - 2.8 In the case that an adjustment or replacement need to be done, please refer to the appropriate section of the *Service operations* chapter (see page 60).
  - 2.9 A complete adjustment verification can be done by referring to page 68, *Adjustment of holders*.

# Visual integrity of heating elements

**IMPORTANT:** Always disconnect the instrument from the power outlet when working with panels removed. 240 Volts inside! Risk of electrocution!

In the K2 Prime, heating elements build a surface oxide layer over the metallic coil, which protects it from degradation and lengthens its lifespan. However, this oxide is extremely sensitive to molten flux.

It is extremely important that no flux ever touch the element. Element life would dramatically be reduced. This is why elements are protected by a clear tube.

Always remove the crucibles from the instrument to weigh components, and make sure that crucible external walls are clean before re-installing the crucibles in their holder.

- 1. Flux spills on elements
  - 1.1 Visually verify that no flux is found on or near the elements.
  - 1.2 If necessary, the clear tube can be replaced independently from the whole element assembly.

#### 2. Wire diameter

2.1 Visually verify that diameter of wire is always the same for all elements. If a section of a heating element exhibits reduced diameter, replace this element immediately.

If an element is found faulty, it should be replaced immediately to prevent any failure of the instrument during a production peak. Elements can be replaced in pairs or all at once, according to the situation and the user's preference.

# Element terminal connections

**IMPORTANT**: Always disconnect the instrument from the power outlet when working with panels removed. 240 Volts inside! Risk of electrocution!

- 1. To check the integrity of the element ceramic connectors and terminals, do the following:
  - 1.1 Unplug instrument (240 volts risk of death by electrocution) and let the furnace cool down completely.
  - 1.2 Remove the element connection access door (refer to page 77, *Heating element replacement* for details on this step).
  - 1.3 Visually inspect and verify that terminals of all four (4) ceramic connectors are not overly oxidized. If a faulty connector is found, replace this ceramic connector immediately.

1.4 Using an appropriate tool, verify that all wire terminals of ceramic connectors are well tightened, both on the element side and the wire side. Do not over-torque the screws. Simply verify that they are not loosened.

## Door springs tension check

This procedure is meant to verify that the springs are properly adjusted, to ensure a correct closing of the furnace.

- 1. Unplug instrument and let the furnace cool down completely.
- 2. Closing test:
  - 2.1 With the power off, the door should remain closed.
  - 2.2 Manually open the door by 25 mm [1 inch].
  - 2.3 Let the door close under its own weight.
  - 2.4 If the door does not close, or if it falls down too abruptly, please proceed topage81, Spring position adjustment, to adjust the springs.
  - 2.5 The spring adjustment is correct when the door closes by itself, slowly but without hesitation.

### Furnace cleanliness

**IMPORTANT**: Always disconnect the instrument from the power outlet when working with panels removed. 240 Volts inside! Risk of electrocution!

- 1. To run this inspection, instrument must be turned OFF, unplugged and furnace at room temperature.
  - 1.1 Unplug K2 (240 volts risk of death by electrocution).
  - 1.2 Remove the safety shield by pulling the far ends of its levers apart. That will free the levers from the white PTFE pivot points.
  - 1.3 Remove the furnace door (please refer to page 81, Furnace door removal for details).
  - 1.4 Visually inspect the door and furnace insulation. Verify that no parts are menacing to fall or are presenting severe cracks that could impair functionality.
  - 1.5 All parts that are found faulty should be ordered and replaced as fast as possible.
  - 1.6 Using dust mask, gloves and vacuum cleaner, remove all dust from furnace and door. Pay attention to do not touch the material with the tube, the material is very brittle.
  - 1.7 Re-install door. While fastening its lever arms, hold the door firmly against the furnace to prevent any movement, and possible future door closing issues.

#### 1.8 Re-install safety shield.

## Thermocouple iunction

**IMPORTANT**: Always disconnect the instrument from the power outlet when working with panels removed. 240 Volts inside! Risk of electrocution!

To run this inspection, instrument must be turned OFF, unplugged and furnace at room temperature.

Thermocouple used in instrument is made of platinum-platinum/rhodium (type R) and is normally very durable. In particular, it is impervious to oxidation or damage by heat; only a mechanical incident could damage the junction (i.e. the tip) of the thermocouple.

- This inspection consists in verifying the junction of thermocouple for visible damage.
  - 1.1 Unplug K2 (240 volts risk of death by electrocution).
  - 1.2 If deemed necessary, you can remove the furnace door (see page 81, Furnace door removal for details).
  - 1.3 Visually inspect the thermocouple junction. Verify that no mechanical shock has occurred, which could havecrushed or otherwise damaged the thermocouple junction. If junction is damaged, a new thermocouple should be ordered and installed.
  - 1.4 Also make sure that the ceramic rod of the thermocouple protrudes by about 10 mm from the furnace insulation. If that is not the case, gently push the furnace insulation back against the rear wall of the furnace. (Note that a thermocouple tip standing too close to the insulation will read inaccurately low temperatures, causing furnace overheating.)

### Door Safety switch

**IMPORTANT**: Always disconnect the instrument from the power outlet when working with panels removed. 240 Volts inside! Risk of electrocution!

To run this inspection, instrument must be turned OFF, unplugged and furnace at room temperature.

The door safety switch removes the power to the elements inside the furnace whenever the door opens. This switch is located in the back compartment of the furnace.

- This inspection consists in verifying the switch actuation lever and electrical contacts for signs of corrosion or possible overheating. Indeed, when the switch is toggled, small electrical arcs can be produced inside the casing, which can lead to a failure of the switch due to over-oxidation of the contact surfaces.
  - 1.1 Unplug K2 (240 volts risk of death by electrocution).

- 1.2 Remove the back panel of the furnace (see page 80, Furnace top/back removal).
- 1.3 Locate the door safety switch. Its actuating lever can be released and depressed when manually opening and closing the furnace door.
- 1.4 Look for signs of corrosion on the actuation lever of the switch, and on the electrical contacts.
- 1.5 Also look for signs of blackening of the electrical contacts.
- 1.6 Replace switch if found dubious.

# Linear motion system

**IMPORTANT**: Always disconnect the instrument from the power outlet when working with panels removed. 240 Volts inside! Risk of electrocution!

- 1. To perform functionality verification, do the following:
  - 1.1 Power ON the K2 and select a method that does not heat (e.g. "TEST").
  - 1.2 Access the Special parameters window (see page 47).
  - 1.3 Press on the "^^^" button to slide the holders in the furnace.
  - 1.4 Press the "vvvv" button to slide the holders out of the furnace.
  - 1.5 Repeat these operations 4 or 5 times to verify that no jerking movement occurs.
  - 1.6 If jerking or abnormal movement occurs, open the base of the instrument to access the linear motion system, correct the problem, and then repeat this test until problem has been resolved.
  - 1.7 If movement occurs normally, no adjustment is needed.

# Service operations

This section describes tasks that are performed on a regular basis, and are performed to adjust or repair a malfunctioning system of the instrument.

Should you have any question, or need further assistance, please do not hesitate to contact us (see page 91, *Contacting Katanax*).

#### Warning

**IMPORTANT**: Some of the procedures described in the following pages imply a risk of death by electrocution; those procedures shall be executed only by trained personnel.

### Platinumware holders service

This procedure describes the steps to take in order to remove your K2 Prime platinumware holders. This procedure needs to be done when cleaning, or when replacing the holders or parts of it.

Note that both holders (crucible and mold) can be removed totally or partially. In routine cleaning, only the middle portions would need to be removed; you would not need any tool. However, if more parts are damaged or soiled, then the whole holders must be removed, and tools are needed. Please refer to page 68, *Adjustment of holders*, to properly re-adjust the holders after maintenance.

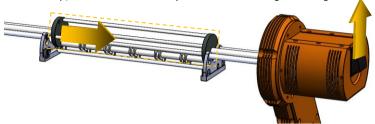
Also, because the crucible holder sits above the mold holder, we recommend removing the crucible holder first, and then the mold holder.

## Crucible holder removal

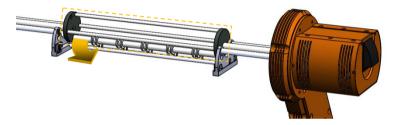
- 1. Crucible holder removal middle section only
  - 1.1 Remove any crucibles and molds from the holders.
  - 1.2 Remove the front ceramic bar of the mold holder.

**IMPORTANT**: The middle section of the crucible holder must remain gently compressed longitudinally at all times, or the ceramic rods, tubes and spacers will fall. Using the elastic band with two clips, originally provided with the complete crucible holder, is recommended for this operation.

1.3 Gently push the left-hand BlackDisk towards the right, to free it from its driving axle. At some point, you will feel that the motion cannot go further, as the assembly reaches an internal stopper. Pull the locking handle up, to allow the assembly to continue moving to the right.



- 1.4 The left-hand side BlackDisk is now free from its driving axle.
- 1.5 Then, while maintaining the crucible holder middle section gently compressed, rotate the left-hand side horizontally towards you, to clear the driving axle completely.



- 1.6 Now pull the middle section to the left, to completely remove it from its axles. A little jiggling may help free the middle section.
- 1.7 You now have in your hands the crucible holder's middle section.



- 1.8 Refer to the following pages to know how to disassemble this subassembly and replace any damaged or flux-soiled parts.
- 2. Crucible holder removal rocking shafts
  - 2.1 Now that the crucible holder middle section is removed, you can disassemble the two ceramic rocking shafts (i.e. the parts that normally hold the crucible holder's middle section). However, unless the rocking shafts are damaged or need cleaning, it is not necessary to remove those parts, which need special care to properly re-install afterwards.
  - 2.2 The rocking shafts are held in collars. These collars can be seen as divided into two halves: the half nearest the furnace holds the ceramic shaft, while the half away from the furnace is holding the motor shaft.

Unless one needs to remove the collars entirely, only loosening the halves nearest the furnace will suffice to remove the ceramic rocking shaft.

# Crucible holder assembly

In this section, you will find essential information needed to properly assemble or repair a crucible holder's middle section. Please follow the instructions carefully, or pouring issues could occur.

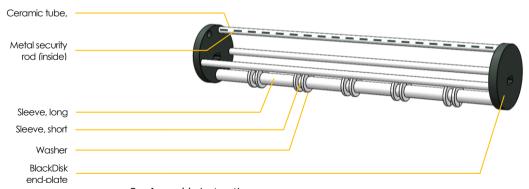
Note that the instructions below pertain to the 6-position holder (part KP6677A, for higher bead throughput). Instructions for the 5-position holder (part KP6077A, typically used only when making solutions) are similar.

#### 1. Parts description

1.1 If your crucible holder is not fully assembled, first check that you have the following parts, available either individually or as pre-packaged kits.

**NOTE**: The information below pertains to 6-position holders; if your holders are configured to accept five (5) crucibles, the quantities and order will be different. Contact Katanax if unsure.

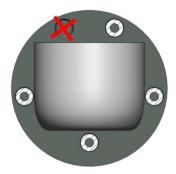
| Description                                  | Part number | Quantity needed per holder |
|--|-------------|----------------------------|
| Crucible holder, full (6 positions)          | KP6677A     | 1                          |
| BlackDisk end-plate                          | KP6277A     | 2                          |
| Ceramic tube, 312.5 mm (bottom/sides/top)    | KP6129A     | 4                          |
| Ceramic sleeve, 41 mm ("long")               | KP0127A     | 6                          |
| Ceramic sleeve, 6 mm ("short")               | KP0459A     | 5                          |
| Ceramic washer (pack of 10)                  | KP0131T     | 1 (pack)                   |
| Metal security rod (hidden inside the tubes) | KP6577A     | 4                          |



#### 2. Assembly instructions

- 2.1 Start by laying out the parts you will need, to identify all parts and make sure all are accounted for. Refer to the table above.
- 2.2 Locate a "BlackDisk" end-plate KP6277A and lay it on a clean surface so that its blind holes face up.

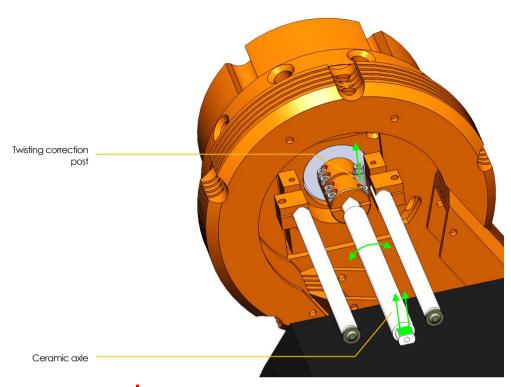
2.3 Insert a "Ceramic tube" KP6129A into each of those blind holes, except one, as shown below. That empty hole will become the front top of the holder (the crucible is shown just for clarity, but must not be installed now).



- 2.4 Into each of the tubes, insert a "Safety ceramic rod" KP6577A.
- 2.5 On the lower ceramic tube, insert:
  - 2.5.1 A "Ceramic sleeve, long" KP0127A, then
  - 2.5.2 A "Ceramic washer" KP0131A, then
  - 2.5.3 A "Ceramic sleeve, short" KP0459A, then
  - 2.5.4 Another "Ceramic washer" KP0131A
- 2.6 Repeat step 2.4 five (5) times.
- 2.7 Add a "Ceramic sleeve, long" KP0127A (still on the lower tube)
- 2.8 Complete the assembly by capping with the other BlackDisk end-plate. Make sure that all holes match properly.
- 2.9 Check for slack on the lower tube:
  - 2.9.1 Once assembled with both BlackDisk end-plates, the stack of spacers and washers of the lower tube should move freely longitudinally.
  - 2.9.2 If more than 2.5 mm longitudinal play is observed, then an additional washer KP0131A shall be added after the last long sleeve KP0127A, to reduce it.

# Crucible holder Installation

- Crucible holder installation instructions
  - 1.1 Before installing the holder middle section, locate the two ceramic axles (see picture below), and make sure that the flats machined on their ends as well as the twisting correction posts face upwards. Manually rotate the axles if that is not the case.



**IMPORTANT:** The middle section of the crucible holder must remain gently compressed longitudinally at all times, or the ceramic rods, tubes and spacers will fall. Using the elastic band with two clips, originally provided with the complete crucible holder, is recommended for this operation.

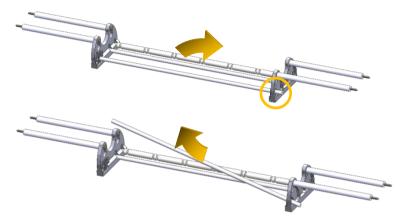
1.2 Take the crucible holder assembly and just follow the removal procedure backwards (see page 60). Do not forget to pull the locking handle up to allow for the extra movement required for easy installation.

# Mold holder removal

- 1. Mold holder removal middle section
  - 1.1 With the crucible holder removed (at least the middle section), the mold holder removal is now possible.
  - 1.2 Lift the front end of the metal mold tabs and gently pull them all out.



1.3 Lift the ceramic rod and slide it about one centimeter through the metal mold holder support opening. Now lift the opposite side to pull out the ceramic rod. Repeat for the two other ceramic rods.



1.4 To remove the metal brackets that normally hold the three ceramic rods previously removed, pull them up and then towards the center of the machine.



- 2. Mold holder removal cantilever supports
  - 2.1 Now that the mold holder's middle section is removed, you can disassemble its cantilever supports (i.e. the parts that normally hold the mold holder's brackets).

**NOTE**: Unless those supports are damaged or need cleaning, it is not necessary to remove those parts, which need special care to properly re-install afterwards. In particular, each ceramic cantilever support should be pencil-marked and identified so that their position is exactly the same after re-installation.

- 2.2 To remove the cantilever supports, loosen the jaw clamps that hold the ceramic rods, and the screw that holds the metal pin.
- 2.3 You can now pull the cantilever rods and metal pins off the main assembly.

# Mold holder assembly

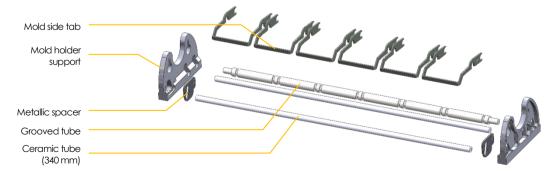
In this section, you will find essential information needed to properly assemble mold holders. Please follow the instructions carefully, or pouring issues could occur.

1. If your mold holder is not fully assembled, or if you have to switch from one mold size to another, first check that you have the following parts, available either individually or as pre-packaged kits.

**NOTE:** The following description relates to 6-position mold holders. If your holder is configured for 5 positions, the assembly will be different. Contact Katanax if unsure.

| Nominal mold dimension (mm) | 30      | 32      | 35      | 40      |
|-----------------------------|---------|---------|---------|---------|
| Complete holder part number | KP6603A | KP6604A | KP6605A | KP6606A |

| Part name                           | Part number  | Quantity required |
|-------------------------------------|--|-------------------|
| Ceramic<br>tube<br>(340 mm<br>long) | KP0137A or<br>KP0137T (pack of 10)                               | 2                 |
| Mold side<br>tab                    | 30mm:KP6730A<br>32mm:KP6732A<br>35mm:KP6735A<br>40mm:KP6740A     | 6                 |
| Grooved ceramic tube                | 30mm: KP6731A<br>32mm: KP6733A<br>35mm: KP6736A<br>40mm: KP6741A | 1                 |
| Mold holder support                 | KP6138A (left side)<br>KP6139A (right side)                      | 1<br>1            |
| Mold holder<br>metallic<br>spacer   | 30mm:KP6630A<br>32mm:KP6632A<br>35mm:KP6635A<br>40mm:KP6640A     | 2                 |



2. Assembly and adjustment are described in the following section.

### Mold holder installation

- With the mold holder components at hand, proceed like the mold holder's middle section removal, but in reverse order.
- 2. However, do not install the front ceramic tube immediately, as it could fall during the crucible holder installation. Wait until the crucible holder is installed, then install the mold holder's front ceramic tube. You may however install the card-shaped metallic spacers, which set the position of that tube.
- 3. Check adjustments as described below.

## Adjustment of holders

To keep your instrument in good working condition, adjustment of the holders must be checked periodically, and each time a part of the holders is replaced.

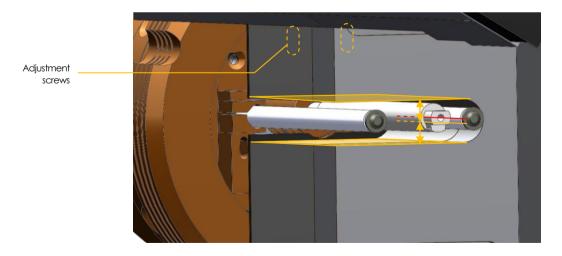
**IMPORTANT**: Incorrectly adjusted holders can cause pouring issues and damage the instrument. Please follow each step in order, to ensure proper adjustment.

Except where noted, all fasteners must be screwed in tightly after adjustment.

Turn the power off before starting this procedure, so that you can manually move the motors when needed. The holders should be fully assembled on the fluxer before starting this procedure.

#### Vertical position of the crucible axles

1. The desired height of each crucible axle is such that it is vertically centered within the furnace slot (i.e. with the same distance to the slot, above and under the axles).



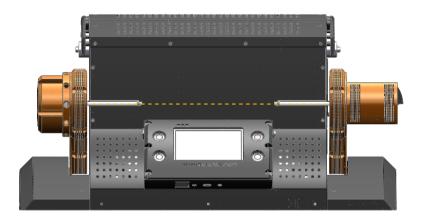
In the picture above, the crucible axle's center (shown in red) is too high and must be lowered (orange) in order to become centered.

An exception is when very thick molds (>2 mm rim/edge thickness) are used; in those cases only, the axles should be positioned 1 mm above the slot centerline.

2. To adjust height, loosen the crucible height adjustment screws, move to correct position and re-tighten screws.

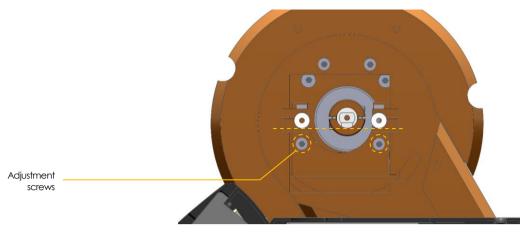
**IMPORTANT:** Make sure that the screws remain parallel with the top edge of the holding arm rectangular cutout. Ideally, you may remove the circular cap covering the motor to have a better view and ensure a more accurate parallelism.

- 3. Repeat with other side's crucible axle.
- Make sure the left and right axles are co-axial one to another. If one axle seems not to point exactly towards the other, please contact Katanax for correction measures.



#### Vertical position of the mold cantilever ceramic tubes

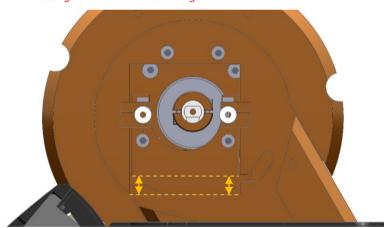
1. The desired height of the mold cantilever tubes is such that their bottom must be at the same height as the bottom of the crucible ceramic axle.



An exception is when very thick (>2 mm rim/edge thickness) molds are used; in those cases only, the mold cantilever tubes' bottom should be positioned 2 mm under the bottom of the crucible ceramic axle.

2. To adjust height, loosen the mold cantilever tube height adjustment screws, move cantilever tube to correct position and re-tighten screws.

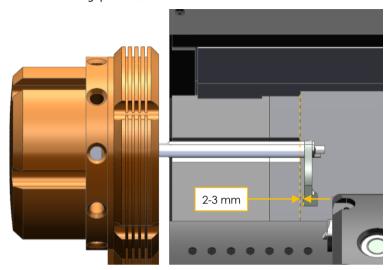
**IMPORTANT:** Make sure that the adjustment plate remains aligned/parallel within the rectangular cutout in the holding arm.



3. Repeat with other side's mold cantilever tubes.

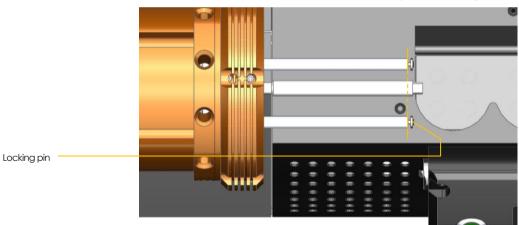
#### Lateral position of the left mold support

1. On the **left** side, the correct position of the mold support is achieved when there is a 2-3 mm gap between the furnace insulation and the mold bracket.



Adjust if necessary, by loosening the jaw's screws, moving the tubes and retightening the screws. Those screws should be on real tight.

2. Ensure that the two left-side cantilever tubes extend by the same length.

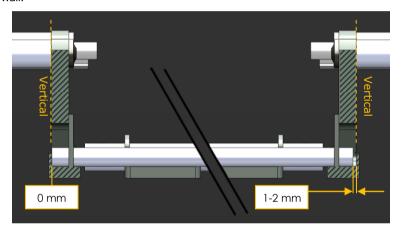


3. The cantilever locking pins may also require adjustment. Their proper insertion position is obtained when the holder bracket is held with near zero play, but can still be manually pulled upwards and removed without tools.

#### Lateral position of the right mold support

- The lateral position of the <u>right</u> mold support is strictly dependent on the position of the left side.
- To obtain the correct position of the right side, install both holder brackets on their cantilever supports. Now, lay the 340-mm tubes in position on the brackets, and then gently move them towards the left until they rest against the bracket stop wall. Make sure that the mold holder brackets hang perfectly vertically and that the thermal expansion system is not compressed.

In that natural position, a 1-2 mm gap should now have been created on the other side, i.e. between the right-hand end of the 340-mm tube and its stop wall.



- 3. Adjust the position of the two right-side cantilever tubes and locking pins to achieve this gap.
- 4. Ensure that the two right-side cantilever tubes extend by the same length.
- Manually open the furnace door and slide the holders inside. There should be a gap of at least 5 mm between the right mold holder support and the side insulation wall. Check position of the holders and of the insulation (which can sometimes shift).

#### Lateral position of the crucible holder

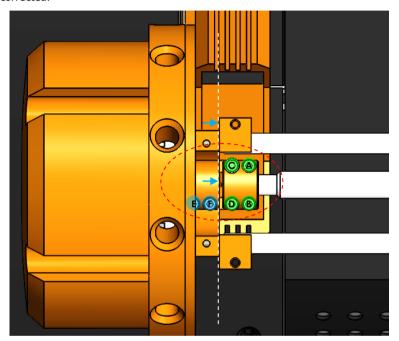
1. The desired position of the whole crucible holder is to be perfectly centered within the mold holder, with the same gap on each side between the mold locking pins and the crucible holder.

The thermal expansion system should not be compressed by more than 1 mm, but it should exert a gentle compression on the crucible holder, so that it has no lateral play (except by pushing the thermal expansion system).

2. To achieve this adjustment, the first step is to turn the fluxer on and let it do the reset motion of the crucible holders.

#### The fluxer shall remain turned ON for the rest of the procedure.

Locate the shaft couplings (circled in red below), which link the motor shafts
to the ceramic axles. Check that the outer face of the slot in each coupling is
coplanar to (i.e. flush with) the holding arm jaws outer face (see blue arrows,
below). If the couplings have just been replaced, this might need to be
corrected.

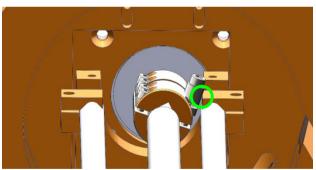


- 4. Then do the adjustment itself, by moving the crucible ceramic axles. There are four (4) screws on the coupling half nearest to the middle of the fluxer, one (1) long and three (3) regulars.
- 5. Unscrew screws A and B by  $1/8^{th}$  turn increments in alternation; once they are quite loose, do the same with screws C and D. This is done to ensure that all facing screws have the same number of turns later on, thus preventing angular dis-adjustment of the axles.
- 6. Carefully push or pull (as needed) the crucible ceramic axle, ensuring that there is no rotational movement.
- 7. Once the correct position is reached, re-tighten the coupling screws in 1/8<sup>th</sup> turn increments: start with screws A and B in alternation, and tighten them fully. Then, do the same to screws C and D (alternate and tighten).

#### Angular adjustment of the crucible holder shaft couplings

Again in this procedure, the instrument must be turned on.

- Remove the crucible holder middle section.
- Enter the "Special parameters" window on the interface, by clicking on the padlock icon at the bottom right of the screen, and enter the code 2206 followed by ENT.
- 3. Set the REF ("reference") twisting correction parameter value to 31°.
- 4. Press U and wait for motion to complete.
- 5. Then press REF button and wait for motion to complete.
- The anti-twisting post of the <u>left</u> side collar should be close to or touching the mold bracket. If not, loosen screw E and F and rotate the collar until they contact, without any lateral motion.
- If the anti-twisting post of the <u>right</u> side collar is not close to mold bracket, manually force the rotation of the former until it hits the mold bracket.
- 8. The ant-twisting posts must be as close as possible without touching the mold holder brackets. The post that is closest to touching will determine the angle to use. Increase or decrease the angle to obtain the best result.
- 9. Then, to verify if the REF parameter is optimally adjusted, press the "U" button and wait for reset operation of the crucible holder to complete, then press the "REF" button. The crucibles holder will rotate towards the back and stop at the set REF angle. In this position, the anti-twisting posts mounted on the left and right orange collars must be in the closest position possible without touching the mold holder orange brackets. (One side may exhibit a larger gap.)



- 10. If one of the posts hits or touches the bracket at this stage, then the REF angle will need to be decreased, and the preceding step needs to be re-done.
- 11. Press the "SYNC" button to verify that 3 full bumps are executed on both sides. This is what one wants when holder is perfectly aligned (not twisted), but sometimes, the number of bumps can be lower, or uneven between right and left sides, in which the REF parameter needs to be increased.

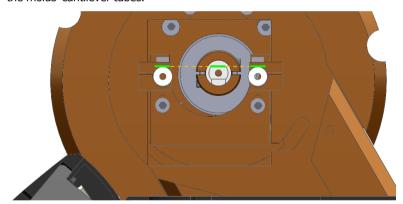
**Note:** A "reset" motion (i.e. crucibles back into straight-up loading position) is done after a twisting correction is triggered by pressing on SYNC. Wait for the reset to complete before attempting other actions on the interface.

- 12. If needed, re-adjust the REF angle to obtain the closest position without contact, and then use SYNC button to check that you correctly obtain 3 simultaneous full bumps on each side.
- 13. Proceed to next procedure to adjust the ceramic axles angular position.

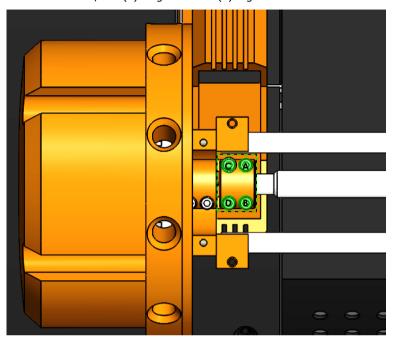
#### Angular adjustment of the crucible ceramic axles

Again in this procedure, the instrument must be turned on.

- Enter the "Special parameters" window on the interface, by clicking on the padlock icon at the bottom right of the screen, and enter the code 2206 followed by ENT.
- 2. Write down the current "Tilt loading" value to restore it after this procedure.
- 3. Adjust the Loading parameter to 0° and press send button.
- 4. Click on the "U" button to force a crucible position reset.
- The machined flat at the tips of both axles should be perfectly parallel with the molds' cantilever tubes.



6. If they are not, locate the shaft couplings, which link the motor shafts to the ceramic axles. There are four (4) screws on the coupling half nearest to the middle of the fluxer, one (1) long and three (3) regulars.



**NOTE:** Using your secondary hand (the one you do not use to hold a screwdriver), firmly grasp the shaft coupling's end that points away from its driving motor (boxed in green). This will prevent the coupling to open fully, hence preventing any unwanted movement.

Unscrew screws A and B by  $1/8^{th}$  turn increments in alternation; once they are quite loose, do the same with screws C and D. This is done to ensure that all facing screws have the same number of turns later on, thus preventing angular dis-adjustment of the axles after adjustment.

Carefully rotate the crucible ceramic axle forward or backward (as needed), ensuring that no lateral motion is applied.

Once the correct position is reached, re-tighten the coupling screws in  $1/8^{\text{th}}$  turn increments; start with A and B in alternation, then do the same to screws C and D.

- 7. Repeat with other side.
- 8. Test your adjustments: press "U" and then "Sync" buttons: the system should complete all motion and come back to 0° position perfectly aligned and not twisted. If not, repeat the procedure until you obtain a perfect result.
- Restore the original "Tilt loading" value, and press "Send" to save it in memory.

#### Final leveling of the instrument

The fluxer comes fitted with three (3) adjustable legs (two in the back corners, and one in the middle front). Once the previous steps are completed, you may use a lightweight spirit level to assess the overall leveling of the mold holders. Screw or unscrew the legs to adjust as needed.

## Heating element replacement

**IMPORTANT**: Always disconnect the instrument from the power outlet when working with panels removed. 240 Volts inside! Risk of electrocution!

There are four (4) heating elements in the K2 Prime, connected in series. Each element is composed of two wire coils running side-by-side.

Two (2) elements are located in the furnace top surface, with the remaining two (2) on the bottom. Each of those elements is fastened to a connector embedded just outside the furnace, on the right-hand side.

To prevent the elements from flexing under their own weight when hot, and to prevent the turns of the coils from touching one another, the elements are wound around ceramic screw-like rods, which is also used to support the left-hand side.

Optionally, the element coils may run inside cleartubing protecting them from flux spills and halogen gases released by non-wetting agents. Those tubes can be removed from the heating element, in order to replace independently either the element or the tube(s) (see page 79, *Element tube replacement* for specific information).

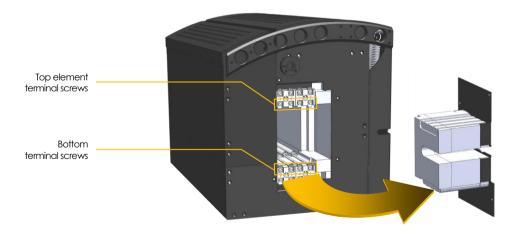
#### Heating element removal

- 1. Disconnect the instrument from the wall outlet.
- 2. Wear surgical-style gloves. They will protect you from the insulation fibers, and will protect the heating element's clear tubes from your skin oil.
- 3. Manually slide the platinumware holders out of the furnace.
- 4. Remove the element side access panel.

4.1 Remove the peripheral screws (circled below).



- 4.2 Manually open the furnace door to assist in the next step.
- 4.3 Place your left hand inside the furnace, with your fingers against the right-hand side furnace wall.
- 4.4 Very gently pull the access panel towards the right. Note that this panel comes with a pretty large block of insulation, which is fragile. That is why your left hand can assist in pushing the insulation block out to the right.



- 4.5 You have now full access to the heating element connectors.
- 5. Proceed to remove one element at a time.

- 5.1 Depending on the element you have to remove, loosen (but do not remove) the corresponding screws (in rectangles above). Support the end of the element when loosening its screws.
- 5.2 Once an element's terminal screws are loosened, slightly pull the right-hand side of the element away from the connector to free its leads from the connector, then pull the element out of the furnace to the right. Support the center of the element when removing the top element, or they will fall and possibly get damaged.

#### Heating element installation

With the side panel removed, you can easily replace any heating element.

- 1. Make sure the instrument is still disconnected from its power outlet.
- 2. Wear surgical-style gloves. They will protect you from the insulation fibers, and will protect the heating element's clear tubes from your skin oil.
- 3. Carefully insert the new element through the opening on the side of the furnace.
- 4. The end of the elements opposed to the terminal side has a flat tab, which must be carefully inserted into the grooves already present in the furnace lefthand side wall. You might want to open the door to locate the grooves and not risk damaging the insulation.
- 5. Push the terminal ends of the element into the connector while strongly retightening the connection terminal screws.
- 6. Gently re-install the access panel with insulation to the rest of the furnace.

#### Element tube replacement

The following section describes the procedure to follow when installing or replacing the clear tubes that can be used to protect the elements.

**IMPORTANT:** Always use clean gloves to manipulate new tubes.

- 1. Remove the heating elements, as described in the previous section.
- 2. Before pulling the tubes off, it may be necessary to gently bend the leads, so that they will slide freely inside the tubes.
- 3. The bending must done just so that the end of the leads comfortably slides through the tubes (see picture below).



- 4. Pull the tubes away, to remove them.
- 5. Slide the new tubes in.

- 6. Gently straighten the leads, back to their original position.
- 7. Re-install the heating elements, as described in the previous section.

#### Furnace top/back removal

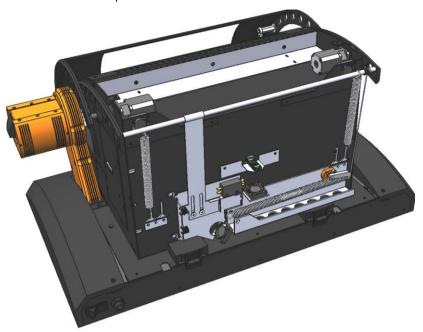
In order to access the door opening springs, the door safety microswitch and the thermocouple bracket, the top/back of the furnace must be removed.

**IMPORTANT:** Always disconnect the instrument from the power outlet when working with panels removed. 240 Volts inside! Risk of electrocution!

- Disconnect the instrument from the wall outlet.
- 2. Locate and remove the screws, as indicated below



#### 3. Remove the back panel.



## Spring position adjustment

This explains how to adjust the furnace door springs to a proper tension.

**IMPORTANT**: Always disconnect the instrument from the power outlet when working with panels removed. 240 Volts inside! Risk of electrocution!

- 1. Disconnect the instrument from the wall outlet.
- 2. Remove the furnace back/top panel (see page 80, Furnace top/back removal).
- 3. Open the door manually and hold it fully open.
- 4. Be careful in the next step, because springs are guite strong.
- 5. Adjust the lower position of the springs by re-attaching them to a different position on the spring adjustment hook. Always adjust left and right springs symmetrically.
- 6. Test the new spring adjustment, as described at page 57, *Door springs tension*.

## Furnace door removal

Removing the furnace door can be necessary for cleaning or maintenance operations.

**WARNING**: Heating elements are powered at high voltage. Always disconnect the instrument from the wall outlet before removing door. When no door is present, safety interlocks are bypassed.

- 1. Disconnect the instrument from the wall outlet.
- 2. Lift the safety shield securely up/backwards.
- 3. Ask for the assistance of a helper.
- 4. While the helper is maintaining the door lever, remove its three (3) front screws.



- 5. With the helper now maintaining both levers, remove other side's screws.
- 6. Have your helper slowly accompany the levers' upwards movement until the levers stop.

#### Furnace backwards flipping

To access components located in the base, it is necessary to flip the furnace to the back:

- 1. Disconnect the instrument from the wall outlet.
- 2. Lay the instrument on a surface large enough to accommodate the back face of the furnace when you will flip it back.
- 3. Manually open the door, and pull the platinumware holders out of the furnace.

4. Remove the two screws that fasten the furnace to its base. Those screws are located just behind the orange sliding parts (see example below).



5. Slowly flip the furnace to the back, about its hinges. Be careful: when the furnace is tilted to the back, the door will want to spontaneously open, because of its springs. Also, the safety shield might suddenly rotate.

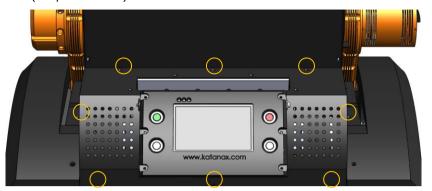
### Blower box removal

In order to access the mold cooling blower or the optional solution agitation module, it is necessary to remove the blower box with LCD screen.

**IMPORTANT**: Always disconnect the instrument from the power outlet when working with panels removed. 240 Volts inside! Risk of electrocution!

- 1. Disconnect the instrument from the wall outlet.
- 2. Move the platinumware holders in the furnace and open the safety shield.

3. Remove the blower box screws: three (3) on the front, three (3) on the top towards the rear, and two (2) on the sides where the linear motion system runs (see picture below).



## Base compartment access

To clean or maintain the linear motion system that pulls the platinumware holder in and out of the furnace, one must open the base of the instrument.

**IMPORTANT**: Always disconnect the instrument from the power outlet when working with panels removed. 240 Volts inside! Risk of electrocution!

- 5. Disconnect the instrument from the wall outlet.
- 6. Flip the furnace backwards (see instructions at page 82).
- 7. Remove the screws from the base top panel (see picture below).



8. Gently pull the cover off.

### Linear carriage

While some installations will never see the need to lubricate the linear motion system, here is how to proceed, should the need arise.

**IMPORTANT:** While it may be tempting to lubricate the tracks, it has been shown that this only attracts dirt. Only the carriage should be lubricated, following the method described below.

- 1. Follow the instructions to access the base compartment (see page 84).
- 2. Locate the two tracks (rails) fastened to the bottom of the unit's casing.
- 3. Wipe down the tracks with a clean, dry rag. Do NOT lubricate the tracks.
- 4. Using an appropriate grease gun, inject lithium-based grease into the grease nipple of each carriage. (The carriages are recirculating ball bearing devices that glide onto the tracks. On one end, they are each fitted with a special nipple designed for lubricant injection.)
- 5. Manually slide the carriages back and forth several times. This will spread the grease inside the carriages.
- 6. The result should be a smooth, continuous motion of the carriages.
- 7. In the unlikely case that you feel there is a need to adjust the belt tension, adjust the position of the front motor so that the tension in both belts is the same, and the tension is such that the belts are just under tension when the carriages reach either end of the stroke.

# Firmware and program transfers

### Preset programs

This section lists the methods that are factory programmed into the K2 Prime. It is not possible for the user to alter or delete them.

Note that Katanax cannot be held responsible for any damage to platinumware or fluxer incurred by the use of those methods. Especially, samples containing unoxidized material should be fused with extreme care. If unsure, please contact Katanax. We will be glad to prepare an adapted method for your specific sample.

| Name     | Applicable to  | Description   |
|----------|--|---|
| Oxide    | Cement, glass,<br>ceramics, petrochemical<br>catalysts, most<br>geochemistry samples<br>and general oxides | Most common fusion method. Must only be applied to fully oxidized material.   |
| Metal    | Samples containing un-<br>oxidized material  | Applicable to metals that can be attacked by acids or bases. This method has a low-temperature oxidation period, in which the solid oxidizer attacks the sample. Fusion is then completed like an oxide.  |
| Solution | Oxidized material to be analyzed by wet method.  | Is normally used with lithium metaborate.   |
| Peroxide | Samples containing<br>metals, often precious,<br>which are attacked in<br>peroxide or peroxide<br>fluxes   | Low-temperature fusion that does not pour. Crucible is removed with tongs at the end, cooled a little on a metallic surface, then immersed into acid. Platinum crucibles are normally not used with this method, as the flux would damage the crucible. |
| Anneal   | Particularly unstable samples that burst or crack  | Provides the user with a template to reheat the mold after casting, and relieve thermal stresses.   |

| Name    | Applicable to                           | Description  |
|---------|---|--|
| Ramping | Samples that require slow heat-up slope | Example of the ramping feature. (See page 40, <i>Ramping</i> for details on how to use this feature in your custom-designed programs.) |

## Backup or restore by USB

Once your favorite methods are developed and optimized, you might want to store them on a USB flash drive, especially before upgrading the firmware of your instrument. This can also be useful to transfer fusion programs among several fusion instruments.

Alternately, you might have received from Katanax a firmware upgrade that you want to install on your instrument.

#### Backup fluxer to USB drive

- 1. To start the process, simply insert a USB flash drive into the USB port of the fusion machine. A dialog will automatically pop up, after a few seconds.
- 2. Touch "Upload".
- 3. A dialog will appear, requesting a password and what data to upload. The password is "111111" (six times the digit one). You may have to drag the dialog window to the left, to show the keyboard and then type the password.
- 4. Select "Upload Project Files" if you want to backup the firmware (i.e. the instrument's operating system).
- 5. Select "Upload History Files" if you want to backup the fusion programs' parameters.
- 6. Touch "OK".
- 7. Now, choose where you want the data to be written. Double-click on "USBDISK", then click on its sub-directory, named "disk\_a\_1".
- 8. Click "OK" to start the transfer to the USB drive. The screen will black out, and the fluxer will re-boot.
- The firmware or the programs are now saved, and you can remove the USB drive.

#### Restore programs, firmware or upgrade firmware

- 1. If you want to restore a backup that you have made yourself, simply insert the USB drive that contains you backup files, into the USB port of the fluxer.
- If you have obtained a new firmware by email, extract the directory structure and files you have received in the root directory of a blank USB drive. Then, insert the USB drive into the instrument's USB port.
- 3. A dialog will automatically pop up, after a few seconds.
- 4. Touch "Download".

- 5. A dialog will appear, requesting a password and what data to download. The password is "111111" (six times the digit one). You may have to drag the dialog window to the left, to show the keyboard and then type the password.
- 6. Select "Download Project Files" if you want to restore or upgrade the firmware (i.e. the instrument's operating system).
- Select "Download History Files" if you want to restore the fusion programs' parameters. Note that this will erase ALL fusion programs currently stored on your fluxer.
- 8. Touch "OK".
- Now, choose where you want the data to be fetched. By default, you will have to double-click on "USBDISK", then click on its sub-directory, named "disk\_a\_1". If the data was created in another (sub-)directory, you will need to browse to reach it.
- Click "OK" to start the transfer to the USB drive. The screen will black out, and the fluxer will re-boot.
- 11. The new firmware or programs are loaded, and you can remove the USB drive.

# Technical specifications

#### **Electrical**

#### **Physical**

<sup>\*</sup> Refer to page 8 for the difference between a regular and an "HX version".

## Warranty

All Katanax instruments have been carefully inspected and tested before shipping and are warranted to be free from defects in parts, material and workmanship for a period of one (1) year from date of shipment. Ceramic parts may exhibit small cracks developed under heat, and will not be considered defective unless this situation impairs functionality of the instrument. Original heating elements, crucible holder assemblies and mold holders are warranted for a period of six (6) months against defects in parts, material and workmanship; however, damage to these parts by oxidation shall be considered normal wear and does not constitute a defect.

During the warranty period, Katanax guarantees the product against defective workmanship and material, provided the equipment has been installed according to the manufacturer's instructions. This warranty does not apply to any product which has been altered, damaged, tampered with, or subjected to misuse or abuse including substituting parts or accessories of other manufacturers without the written consent of Katanax. Minor adjustments are not covered by warranty.

Katanax disclaims any responsibility for misuse, misapplication, negligence or improper installation and maintenance of equipment. Katanax makes no warranty or representation regarding the fitness for use or the application of its products by the purchaser.

Katanax is not liable for costs incurred in installation, removal or unauthorized repair of the product or for damage of any type, including incidental or consequential damage.

At its option, Katanax will repair or replace any defects that are exhibited under proper and normal use. Replacement parts are covered for one month after shipping. All customs- and freight-related charges are customer's responsibility: items returned to Katanax for any reason shall be via freight prepaid, while parts sent to customer will be either sent collect, or shipping charges will be invoiced.

Katanax reserves the right to make changes in the design or to make additions or improvements with respect to its product without incurring any obligation to modify or install same on previously manufactured products.

# **Contacting Katanax**

Katanax sales and technical staff can be reached at the following address:

Katanax inc. 2022, Lavoisier, suite 100 Quebec City, QC Canada G1N 4L5

Tel.: +1 (418) 657-6201 Fax: +1 (418) 657-6203

E-mail: info@katanax.com Web: www.katanax.com

When contacting us, kindly have the serial number of the instrument at hand.

Customers are invited to visit our web site regularly, since useful information is periodically added.

An illustrated online parts browser is also available; kindly contact us to register.