UVM Cosmogenic Laboratory

Laboratory Safety Manual

Delehanty Hall Room 326

Quartz Purification Protocol

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Individuals responsible for lab and lab safety

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Senior Technician Jennifer Larsen jlarsen@uvm.edu 802 656 3519 (office) 802 655-4226 (home) The mineral separation laboratory is where minerals, present in rock and sediment, are separated based upon their physical and chemical properties. Strong magnets remove some minerals. Dilute acids are used to etch away more easily dissolved minerals. Heat is used to combust carbon-bearing materials and heavy liquids are used to separate minerals based on their density. Various mineral acids, as well as tungstate-based heavy liquids, are used in the laboratory. Access to the laboratory is restricted to personnel specifically trained in the use of concentrated mineral acids and in proper waste disposal.

For most samples, the goal of work in the mineral separation lab will be the production of very clean and pure quartz. Many years of work has shown that 10-Be is more easily and reliably extracted from quartz with low impurity levels. Patience, meeting or exceeding the stated processing times, and not skipping steps or overloading bottles will result the purest quartz possible.

This manual contains the following sections:

- 1. **Personnel capability checklist for laboratory procedures** which must be filled out for each person working in the lab. This checklist demonstrates that you have been trained in all aspects of laboratory methods and safety.
- 2. **Safety Considerations and Equipment** including: laboratory access, safety equipment in the lab, laboratory safety training protocol, and a list of chemicals used in the lab, monthly self inspection instructions.
- 3. **Sample Processing Protocols including:** Washing with water to remove fines, drying sample, hot 6N HCl etching in Teflon, sample leaching in HF and HNO₃, density separation, combustion, ultrasonic sample leaching in HF and HNO₃, final magnetic cleaning, checking quartz quality, tube labeling.
- 4. Chemical ordering, waste disposal, and chemical storage including: how to order acid, when to order acid, acid disposal procedure, how to store chemicals, how to fill our waste tags, how to get waste picked up, and disposing of empty chemical bottles.
- 5. **Materials Safety Data Sheets:** These are basic chemical information sheets for all chemicals used in this laboratory.
- 6. **Operating Manuals**: These are manuals for the ultrasonicators, the furnace, and the acid dispensers.
- 7. Kohl and Nishiizumi (1992): This is a paper describing the method we use and justifying particular acid concentrations and sample size ratios

Section 1 - Personnel capability checklist for laboratory procedures

During training, each person who will be working in the lab needs to complete this list with initials as they master skills and gain knowledge. Only after this list is complete, can a new user work alone in the lab.

	Item	Date	Personnel initial	Trainer initial
	Understand importance of producing clean quartz			
	Understand access limitations for lab			
	Complete on-line safety courses			
	Complete in-person safety courses			
E	Review incident flow chart			
H	Know which chemicals are used in the lab and have read			
S	the MSDS for all chemicals in the lab			
Q	Understand chemical storage and movement procedures			
Z	Understand acid bottle waste disposal procedure			
	Understand CaCO ₃ waste disposal procedure			
Ž	Understand use of all personal protective gear in			
	laboratory			
V	Understand where lab coats and non lab coats are hung			
K	Understand policy on open toed shoes			
H	Understand policy on food in the lab			
0	Know location of eyewash and safety shower and know			
T	how to use each			
RA	Know when and how to test eyewash			
E	Know where Calcium Gluconate cream is located and			
E	how and when to use it.			
5	Know where fire alarm, red phone, and fire extinguisher			
	are and how and when to use them			
	Read and understand the monthly self inspection cards			
	Understand how and when to test ultrasounds			
	Able to launch sample database			
Ę	Understand how to enter sample information in database			
L	Understand how to create a batch in the database			
Σ	Understand the importance of tracking every action in			
Y	the database			
	Understand how and when to enter information in the			
	sample tracking database			

NAME: _____

	Item	Date	Personnel initial	Trainer initial
	Know where trays and beakers are kept			
	Understand how to wash samples			
	Understand importance of double labels			
9	Understand where and how HCl is stored			
	Understand how to add 6N HCl to samples			
E	Understand how to mix 6N HCl safely			
Ľ	Understand how and why to fill ultrasounds with DI			
Ξ	Understand how to set timers			
Γ	Understand how to rinse reload samples and do final			
C	wash to prepare samples for drying			
jului	Understand how to test pH of wash water			
	Understand how oven works			
	Understand how to dry and label samples after HCl			
	Understand safety gear and precautions needed to use			
	concentrated HF and HNO3 including gloves smock			
	and face shield			
	Understand where HF and HNO3 are stored			
	Understand how why and how often to check bottles for			
	stress cracks			
	Understand how to label bottles and the importance of			
75	labeling lids			
ž	Understand how to lift bottles safely			
H	Understand how to use digital balance			
U	Understand how to date new bottles			
	Understand how to enter sample weight into database			
H	Understand how to dispense HF and HNO3 safely			
Ţ	Understand how and how much to fill bottles			
	Understand how to indicate that etches have been done			
3	in the sample database			
Õ	Understand how to rinse bottles between etches			
	Understand how to rinse bottles after last etch			
	Understand how to condense low quartz samples into a			
JU	single bottle for drying			
L.	Understand how and where to dry samples			
Η	Understand how to label tubes to indicate that etching is			
	complete			
	Understand how to transfer sample to tubes and the			
	importance of using new paper for each sample			
	Understand the importance of having only one sample		1	
	out of the oven at any time.			
	Understand the importance of and process for cleaning			
	bottles after use			

	Item	Date	Personnel initial	Trainer initial
	Understand when and why burning is important and			
	when it should not be done			
	Understand where crucibles are stored			
5	Understand how to label crucibles and how to fill in the			
	Turnace layout map			
Z	Understand now to load the crucibles and the furnace			
5	Understand now to turn on the furnace			
B	Understand now to return the samples to their vials and			
	burned			
	Understand how to enter burning step in the database			
	Understand when density separation is needed.			
	Understand importance of recycling LST			
	Understand how to change LST density			
	Understand why LST is stored in two different bottles			
Z	Understand why there is a quartz chip and how it is used			
10	Understand what the stopcock is and how to grease it			
L	Understand how and when to hook up the vacuum			
R	system			
	Understand how to load filter paper			
SEI	Understand how to separate quartz from mafic minerals by adjusting density of LST			
	Understand how to extract the purified quartz			
	Understand how to reclaim the dilute LST			
NS	Understand why overnight hotplate operation is not			
)E	allowed			
	Understand how to clarify cloudy LST			
	Understand how to make purple LST clear			
	Understand how to transfer clean quartz back into the			
	proper tube and how to label the tube to indicate that			
	mineral separation has been completed			
лг	Understand that 48-nour etching is done in 1 liter bottles			
101	Understand that 48-hour etching uses weaker acid			
8-1	Understand how to label tubes after 48 hour etching			
4	Understand how to do final magnetic cleaning			
\mathbf{S}	Understand how and why we keep an acid tally			
T	Understand how and when to order more acid			
VIV	Understand how and when to ask Paul about ordering			
ER	more LST			
E	Understand how fill out waste tags			
I A	Understand how old sample bottles are disposed			

Section 2 - Safety Considerations and Equipment

1. Lab access - Room 326

Access to the mineral separation laboratory is restricted to faculty, staff, students, and visiting scientists and students who have been trained specifically as outlined in the safety training protocol below. Keys will be issued on request (and with a deposit paid to the department) to those meeting the training criteria.

2. General Laboratory Practice

a. Personal protective equipment must be worn at <u>all</u> times when dealing with acids. This includes goggles, gloves and rubber smocks. The face shield must be used when handling concentrated HF and HNO₃.

b. No open toe shoes or sandals are allowed. Rubber boots are available in the lab.

c. Extra clothing should be placed on the coat rack behind the door.

d. Personal bags/backpacks should be placed in the rolling bin beneath the desk.

e. No food or drink should be stored or consumed in the lab.

3. <u>Safety equipment in lab</u>

Personal Protective Equipment - MUST be worn at all times when working with chemicals.

Goggles – Stored in drawer to the right of the plastic hood, goggles should be donned whenever any work involving chemicals of any sort is done in the laboratory – no exceptions.

Gloves – Various gloves are stored in the lab and used for different tasks. When using HF, double gloving is preferred with a thin under glove and a thick, black and blue over glove.

See Ansell 7th Edition Chemical Resistance Guide pdf (http://www.ansellpro.com/download/Ansell_7thEditionChemicalResistan ceGuide.pdf) for further details of the chemical resistance of gloves (a copy is kept in the mineral separation lab, room 326).

Boots – Rubber boots are stored under the lab coats and must be worn if the user does not have closed toed shoes.

Face Shield – The face shield hangs on the wall of the lab and should be used in addition to goggles whenever concentrated HF or HNO_3 are dispensed.

Eyewash – Located by the laboratory sink, it should be rinsed weekly and the rinse sheet initialed and dated. It should be used immediately if any chemicals are spilled on or near the eye.

Safety shower – Located by door, the shower should be used in acid is spilled on your body (see incident flow chart). The shower is tested several times a year by the departmental technician.

Incident flow chart – Posted on the wall inside and outside the lab, this chart details responses to chemical hazard incidents that could occur in the lab. It should be studied and is included in this document.

Calcium Gluconate cream – Stored over the sink, this cream should be applied to any suspected HF exposure immediately as one is proceeding to the emergency room.

First Aid Kit – Is located next to the lab door and can be used for minor injuries.

Fire extinguisher – Is located by the door to the lab and can be used to extinguish minor fires.

Fire Alarm – Is located in the hall outside the lab near the stairs and should be used for major emergencies including any fire as well as major chemical spills.

Red Phone – is located in the hallway immediately opposite the laboratory and when picked up will directly dial UVM police services. Use this phone in any kind of emergency.

Spill Kits – located on the shelf to the left of the door as you enter the lab. Only to be used for very small spills (<10 ml). It is preferred that ESF is called to clean up all spills outside the fume hoods. The spill kits are NOT TO BE USED for an HF spill.

4. <u>Safety training protocol</u>

Before working unsupervised with hazardous materials, all personnel must complete the following on-line safety training.

The following three on-line courses need to be taken and are available at: <u>http://esf.uvm.edu/courses/</u>

Chemical Safety in the Laboratory Laboratory Waste Disposal Pollution Prevention in the Laboratory

As soon as is practical, all personnel must complete the following *in person* safety classes. These courses are offered at regular intervals and require pre-registration.

Emergency Response for Laboratory Workers (1.5 hours) Using Fume Hoods and Biological Safety Cabinets (45 minutes) Van Safety Training (3 hours)

5. <u>Chemical list</u>

- **a.** Hydrofloric Acid (HF) Used for acid etching of samples to remove almost all other minerals but quartz. Supplied in 2.5 liter plastic jugs. Dispensed in hood using dispenser. Stored in dedicated secondary containment under hood in vented cabinet. One bottle with dispenser attached is stored in the plastic hood. Diluted to 1-2% for etching.
- b. Hydrochloric Acid (HCl) Used for removing Al, Fe, and carbonate coatings from grains as well as dissolving iron filings from grinding process. Supplied in 2.5 liter glass jugs. Dispensed by pouring. Stored in dedicated secondary containment under hood in vented cabinet. Dilutes to 6 N for etching.
- c. Nitric Acid (HNO₃) Used along with HF to etch samples; keeps fluorides in solution. Supplied in 2.5 liter glass jugs. Dispensed in hood using dispenser. Stored in dedicated secondary containment under hood in vented cabinet. One bottle with dispenser attached is stored in the plastic hood. Diluted to 1-2% for etching.
- **d.** Lithium Polytungstate (LST) Used for density separation. Density can be changed by the addition of DI water. Supplied and stored for reuse in 1 liter plastic bottles. Stored under the mineral separation hood. Extremely expensive. Must be conserved and recycled.
- e. Hydrogen Peroxide (H_2O_2) Used to regenerate LST and remove color. Supplied and stored in plastic bottle. Stored in secondary containment under the mineral separation hood.

6. Monthly self inspection

UVM mandates that we conduct a monthly self inspection of all laboratories to identify and remediate hazards before they cause accidents and injuries.

On or about the first of each month, Jennifer Larsen, senior technician, will inspect the mineral separation laboratory and note on the self inspection cards any outstanding issues. She will email her findings to all students, faculty and staff using the mineral separation facility as well as lab supervisor Bierman. We expect that any deficiencies noted by Larsen will be addressed as quickly as is possible.

Please familiarize yourself with the self-inspection sheets which are hanging by the door of the lab.

7. Ultrasound filling and water evaporation

The ultrasounds require filling on a daily basis when they are being used and refilling before use if they have been left idle. During use, the heated water rapidly evaporates. To minimize this evaporation, we use small floating plastic balls which cover the water surface and/or plexiglass covers in which beakers and tubes sit.

When using any ultrasound, ensure that before it is started and at least every 24 hours, you check for proper water level. The proper water level is just below the gasket at the top of the tank. On older ultrasounds and baskets, this will be identified by deep staining. Overfilling the ultrasounds will lead to moisture seeping past the gasket into the electronics. Letting the water level drop too low will cause both the heating and sonicating elements to fail. In both case, repairs are costly and more importantly take the better part of two weeks since the unit needs to be sent away.

8. <u>Ultrasound performance check</u>

The ultrasonicators that are the backbone of the lab can run for years if properly cared for. However, parts do fail over time and if the sonicators are not working properly, quartz purity will suffer. The ultrasounds should be tested monthly along with the eyewash and the results recorded on the ultrasound clipboard.

The ultrasounds should both heat and sonicate. Heating should raise the temperature of the water in the tank to between 60 and 70 degrees C after an hour or two of operation.

Sonication should be checked monthly by using an etched slide. soap and a pencil (see the Branson operating manual at the end of this document for full details). On the next page is a check sheet that should be initialed every time the sonicators are checked. It should be hanging on a clipboard in the lab.

- 1. Mix up a soapy solution of hot water in a 1-liter bottle using the lab detergent
- 2. Use #2 pencil to make a bold X on a frosted slide (there is a box of slides in a drawer near the ultrasounds)
- 3. Make sure tanks are filled
- 4. Run ultrasounds for at least 10 minutes with bottle of soap filled water sonicating.
- 5. Hold the end of the slide, immersing it in the soapy solution.
- 6. The pencil mark should come off the slide within 10 seconds. If the slide does not come clean, see Paul

	US number							
Date	#1	#2	#3	#4	#5	#6	#7	#8
				10	5			
							6	
								50
				00				

Please fill in your initials to indicate that unit has passed soap test



Section 3 - Sample processing protocols

Overview of Mineral Separation Laboratory Processes

- 1. Enter samples into the database.
- 2. Create batch in database.
- 3. Sample Preparation.
 - **a.** $2 \ge 6$ N HCl etches.
 - **b.** Sample leaching 3 x HF/HNO₃ etches (ultrasound).
 - c. Burn samples in muffle furnace (unless 21-Ne analysis is needed).
 - d. Density separation (if necessary).
 - e. Sample leaching 48 hour etch.

An acid dumping log will be produced automatically from entries in the database. It is CRITICAL therefore to complete database entries after each step in the mineral separation lab.

The following are sample processing protocols that have been designed to yield purified quartz. They have been developed by trial and error over the past 15 years and represent a conservative approach most likely to yield clean quartz. For some projects and some samples, procedures may need to be modified after experimentation. The goal is to obtain a final yield of ~ 20 g of clean quartz.

Manuals for the equipment in the lab are placed in plastic folders next to each piece of equipment. They are also on-line at uvm.edu/cosmolab

1. Entering samples in the database

Each sample needs to be entered in the on-line cosmolab database before it is processed. Data entry is usually done by the person who collected the sample and includes: sample ID, sample site photographs, sample information including rock type, collector, and GPS location. Scanned field notes and sample sheets also need to be uploaded.

2. <u>Create batch in the database</u>

Create a new batch and enter sample names to be processed.

3. Sample preparation

Before working with acid appropriate personal protective equipment should be worn – goggles, rubber smock, thick neoprene gloves (with vinyl or latex under glove), rubber boots.

To maintain sample integrity and avoid confusion between samples, it is imperative that you work with only one sample at a time.

a. 6N HCl etching in Teflon (do this 2 times for 24 hours each)

Purpose: To remove grain-coating oxides from grains and dissolve carbonate material.

Processing Time: 2 x 24 hours

Lab ware and materials needed:

Sieved 250-500 or 250-850 µm grain-size fraction of samples to be processed. 500 ml savillex teflon beakers. 6N HCl. Labeling tape and sharpie.

Procedure:

- i. Bring up live batch in the database.
- ii. Pull out eleven or twenty two 500 ml Savillex Teflon beakers and lids designated for etching. Check each one to see that it is clean of quartz grains. Rewash if necessary. No need to dry. Collect 11 or 22 ground and sieved samples. Make two labels for each sample.
- iii. Label beaker and lid with tape. Fill each beaker halfway with sample material from one sample (USE 500-800 μm FRACTION IF IT IS SUFFICIENT, add 250 to 500 material only if needed) and write sample name on tape on beaker and lid.
- iv. Dump contents of beaker into a broad shallow drying tray. Place tray in the sink and turn on the water gently. Use the gentle stream of water and stir. You should adjust the stream so that only the finest material is washed out of the tray as you stir. Continue stirring until the rinse water is clear and the mica no longer floats out of the samples (usually a few minutes). If the sample is mica-rich, this is the most effective means of mica removal. Be patient and keep washing until no more mica floats off; washing now will pay off later. Drain the sample and transfer sample back to the beaker. Fingers may be helpful, just make sure they are quartz free. Repeat for all samples. FOR MICA RICH SAMPLES, use a large basin and plenty of water attempting to float off the micas.
- v. Transfer beakers to hood one at a time. Add a little HCl and see if there is a reaction (if so, wait until it subsides, the reactant is carbonate, most likely from samples collected in limestone terrain or arid region soils). Add additional 6 N HCl sufficient to cover sample by about 2-3 cm. STIR with stir rod. Place cap firmly on beaker. Put beaker into Plexiglas cover on the ultra sound. 6 N HCl is a 1:1 mixture of concentrated acid and DEIONIZED water. Always add acid to water for safety. Mix up acid in bottle found under the hood.
- vi. Fill ultrasound just below gasket level with DEIONIZED water. DO NOT OVERFILL. Set timer for 24 hours and etch. More etching will not hurt the samples but running ultrasounds low on water will cause large repair bills.
- vii. After at least 24 hours of sonication, drain beakers into the plastic hood sink. Rinse in hood at least three times with DEIONIZED water stirring with clean stir bar all the way to the bottom of the beaker. Thorough washing is important. Refill the beakers with HCl and repeat with a second 6N HCl etch.
- viii. After the second 24 hour HCl etch, rinse the sample twice in the beakers, dump the sample into a small drying tray and MAKE SURE TO TRANSFER THE TWO LABELS TO THE TRAY. Wash the sample repeatedly in the tray (5 to 10 times), test the pH of the rinse water (should be at least 7).

- ix. Decant as much water as possible and transfer to the drying oven checking again to make sure the tray is labeled.
- x. As soon as material is dry, transfer sample to labeled plastic bag labeled to indicate that material has been HCl etched. Store the bags in the labeled drawer that is assigned to your samples.
- xi. Check off HCl etch in the database.

b. Sample leaching in HF and HNO₃

SAFETY PRECAUTIONS

Both concentrated HF and HNO₃ are potentially dangerous acids. Under no circumstances should either acid be allowed to contact the skin, nor should the fumes be inhaled. These acids should be used ONLY in the fume hood and only if the user is wearing FULL protective clothing including eye protection.

1. ALWAYS WEAR GOGGLES WHEN HANDLING ACID. 2. ALWAYS WEAR FULL RUBBER SMOCK, THICK NEOPRENE GLOVES, AND CLOSED TOED SHOES WHEN HANDLING EVEN DILUTE ACID.

3. BEWARE THAT LIDS MAY NOT SEAL TIGHTLY, LIFT BOTTLE BY BASE.

4. NEVER WORK LATE AT NIGHT WHEN YOU WOULD BE ALONE. 5. RINSE ANY SPILLED OR SPLASHED ACID IMMEDIATELY WITH COPIOUS AMOUNT OF TAP WATER.

6. TIGHTLY CAP HF BOTTLE AFTER EVERY USE. DO NOT LEAVE UNCAPPED EVEN FOR A MOMENT.

7. LEAVE SIGN ON HOOD SO OTHERS KNOW ACID IS BEING USED.

Purpose: To dissolve most everything but quartz. **Processing time:** 3 etches of at least 24 hours each. Total of at least 72 hours.

Lab ware and materials needed:

Ultrasound HCl etched sample. 1 liter HDPE nalgene bottles and lids (12 bottles per ultrasound) for very quartz-rich samples 4 liter PP nalgene bottles and lids (ultrasounds – 3 bottles per ultrasound) for most samples HF/HNO₃ Digital balance. Labeling tape and sharpie.

Procedure

i. Read Kohn and Nishiizumi (the paper that lays out this method). You can find it at uvm.edu/cosmolab/papers or at the back of this document.

- ii. Bring up live batch in the database.
- iii. Pull out 4 liter bottles and lids (most samples) or 1 liter bottles and lids (very quartz rich samples)
- iv. Check each bottle carefully for stress cracks by examining the bottom and the handle. If a bottle has cracks, destroy it and notify Paul reporting the date on the bottle. If you put a new bottle into service, use a sharpie to write clearly and boldly the date on the upper part of the bottle.
- v. Label all bottles PRIOR to adding sample.
- vi. Weigh out between 40 and 50 grams of dried, HCl etched sample into each 4-liter bottle OR weigh out between 20 and 40 grams of dried, HCl etched sample into each one-liter bottle. Weight and date are automatically recorded into live batch database.

vii. First Etch (24 hours)

Ultrasound

- Fill each bottle ³/₄ full with DI water.
- Dispense 50 ml of HNO₃ (two squirts) and 75 ml of HF (three squirts) into each bottle, and screw the lid on tight.
- Place all bottles in the ultrasounds (3 4-liter or 12 1-liter bottles will fit in each one).
- Set the timers behind the ultrasounds for 24 hours. Turn them on
- viii. Check the box for first HF/HNO₃ etch in the database. The date will be automatically entered.

ix. Second Etch (24 hours)

Ultrasound

- After the first 24 hours, take the bottles out f the ultrasounds and stack them on the grey rolling cart.
- In the acid hood, **CAREFULLY** decant the murky water from each bottle into the sink. Refill each with pressurized DI water. After letting them stand for a minute (to let larger sediment grains settle) decant each again. This process works best if you stagger bottles; decant and refill one bottle with another is settling.
- When all the bottles are rinsed, refill them ³/₄ full with DI and add acid exactly as for the first etch. Put them back in the ultrasounds for another 24 hour etch. Reset the timers and turn them on.
- x. Check the box for second HF/HNO₃ etch in the database. The date will be automatically entered.

xi. Third and Final Etch (24 hours) Ultrasound

- Rinse each sample bottle again as above, and reload with acid.
- Place in the ultrasounds for yet another 24 hour etch.
- xii. Check the box for third HF/HNO₃ etch in the database. The date will be automatically entered.

xiii. Final Rinse and Dry Down

- Rinse each sample as with prior steps.
- Particularly for low quartz yield samples, condense the clean quartz from all bottles into one single bottle. Rinse this single bottle 2 to 3 more times.
- Make sure the bottle is labeled, and place on the top shelf in the drying oven over night. The oven is set at $\sim 60^{\circ}$ C.
- xiv. Place dry sample in a labeled 50 ml vial using a separate paper funnel for each sample. It is IMPERATIVE to use new paper for each sample and to clean up

immediately any spilled quartz. Remove only one sample from the oven at a time. Label a vial for that sample. Double check that the label matches the label on the bottle. Note on the vial that it has been HF/HNO₃ etched and the date.

- xv. Store the vials in a rack in the drawer labeled to burn.
- xvi. Weigh the sample using the digital balance. The weight and date will automatically be recorded in the database.
- xvii. Rinse all the plastic bottles and trays carefully with a scrub brush making sure to remove ALL quartz. Use no soap.

IT IS IMPORTANT THAT THROUGHOUT THIS PROCESS CROSS-TALK BETWEEN SAMPLES BE KEPT TO A MINIMUM. THIS MEANS EVERY BOTTLE MUST BE CAREFULLY RINSED BETWEEN SAMPLES. CHECK YOUR BOTTLES BEFORE and AFTER USING THEM FOR BOTH CARRIED OVER QUARTZ AND CRACKS.

c. Burn

Purpose: remove any organic material (coal etc) that survived etching. This step is mandatory for any soil and sediment samples and useful for samples from sedimentary rocks. It is not usually required for crystalline rocks. **Processing time:** 5 hours (run the furnace at the end of the day).

Lab ware and materials needed:

HF/HNO₃ etched samples. 50 ml porcelain crucibles. Graphite pencil. Furnace layout map.

Procedure

The furnace has been programmed for the burn. It will ramp up to temperature, run for 5 hours at 500° C, and then cool down.

- i. Collect HF/HNO₃ etched samples.
- ii. Label the unglazed bottom of each crucible with the sample ID and note its position in the furnace on the furnace layout map.
- iii. Empty each sample into the crucible.
- iv. Place the crucibles in the furnace.
- v. Turn on the power and the furnace display cycles through until it displays the internal temperature. To run the program press the SCROLL button until StAt is displayed. Press the up/down buttons until "run" is displayed. Press the page button to display the actual temperature. The furnace cycle light will come on (orange) and a green light (OP1)will appear in the top left corner of the display next to the temperature.
- vi. To stop the program press the SCROLL button until StAt is displayed, set to "oFF" with the up/down buttons.
- vii. To clear the flashing end on the display press the PAGE and SCROLL buttons at the same time.

- viii. Turn off the power and empty the furnace. Return the sample to the appropriate 50 ml vial using a separate paper funnel for each sample and **note on the vial that it has been burned and the date.**
 - ix. Bring up live batch in the database. Check off burn on the database. The date will be automatically recorded. Store the vials in racks in the drawer labeled to density separation.

d. Density Separation

Purpose: to separate quartz from denser mafic minerals. **Processing time:** Approximately 15 minutes per sample.

NOTE: this step must be done if ANY mafic (dark colored) minerals remain in the purified quartz.

Personal Protective Equipment – lab coat, goggles, vinyl disposable gloves.

NOTE: Per UVM policy, overnight operation of the hotplate used to evaporate water from the LST is NOT allowed. Please make sure that you turn off the hotplate before leaving for the day.

LST is a non-toxic replacement for halogenated heavy liquids. It is unfortunately extremely expensive and an eye irritant. Therefore, gloves and goggles are necessary as is reclamation of as much of the LST as is practical. Such reclamation means that all glassware should be well rinsed and the LST in the rinse liquid be recovered by evaporation. Such recovery is speeded if more concentrated LST is maintained separately from the less concentrated LST during the rinsing and recovery process.

Lab ware and materials needed:

Pyrex beakers LST (lithium polytungstate) (specific gravity: 2.85 @ 25° C) 1000 ml separation funnel 1000 ml Pyrex vacuum flask Filter funnel with rubber stop Filter paper (VWR grade 413, 7.5 cm) Petri dishes for drying sample Quartz density chip Hotplate Vacuum pump Sample DI water Polypropylene stirring rods Ring stand Labeling tape and sharpie.

Procedure

- i. The LST is stored in two different bottles. One bottle contains undiluted LST, the other contains LST at the correct density for the density separation process. To prepare LST to the correct density, add some undiluted LST to a Pyrex beaker. Slowly add DI water, a few drops at a time if the LST is near the correct density for quartz and stir well. Test the density by dropping in a quartz chip. If the LST is at the correct density, the quartz chip will rise very slowly from the bottom of the Pyrex to the surface.
- ii. Grease the stopcock for each of the separatory funnels; place it in the separatory funnel in the CLOSED position. Set separatory funnel in ring stand. Place a vacuum flask beneath each separatory funnel and attach to the vacuum hose line. Fit the filter funnel with rubber stop into vacuum flask and drop in a sheet of filter paper. Wet the filter paper with a little deionized water. Lower the rings supporting the separatory funnels so that the lower tip of the funnels matches the height of the lip of the Buchner funnels (this step prevents splattering of the LST liquid).
- iii. Pour LST (density >2.7) into the separatory funnel using a polypropylene funnel or directly from the Pyrex beaker. Put in enough liquid to cover the sample by \sim 5 cm.
- iv. Pour the sample from the 50 ml vial into funnel. ATTACH A PIECE OF LABEL TAPE TO THE SEP FUNNEL WITH THE SAMPLE ID WRITTEN ON THE TAPE.
- v. Stir the sample and allow 2-5 minutes for the mafics to sink to the bottom. Drain the mafics from the separatory funnel into the filter paper. Do this by opening the stopcock fully and then shutting it completely. Then, open and close the stopcock going smoothly in the other direction to remove grains sticking at the opening. Stir the sample vigorously and repeat process until most of the mafics are removed from the sample (some small additional amount of DI water (drop by drop) may need to be added to allow the smaller and lighter mafics to sink).
- vi. After the mafics have been dropped, turn on the vacuum pump and use the squirt bottle to clean the LST from the mafics and to quicken the filtering of the LST. Once the dripping of the filter into the vacuum flask has ceased or slowed considerably, turn off the pump and remove the filter funnel. Place it into a separate 1000 ml flask to allow it to drip. Pour the LST from the vacuum flask into a Pyrex beaker for dry down on the hotplate (low heat, just above warm). This takes a few hours. Test the density with a quartz chip. When ready, pour the LST into the separation funnel, and drain through the filter funnel, containing new filter paper, with the vacuum on. When filtered, store the LST in the "at correct density" bottle). Dispose of the filter paper containing the mafics into the waste jar in the cabinet under the hood. Place a new piece of filter paper in the filter funnel and dampen it.

- vii. Return the vacuum flask with the filter funnel to the vacuum line. Turn on the vacuum and drain the remaining LST and sample into the filter funnel. Turn off the vacuum. Remove the filter funnel (place it on a separate 100ml flask) and decant the LST from the vacuum flask into the "at the correct density" LST bottle. Replace the flask and filter funnel and attach the vacuum tube. Rinse down the sides of the separation flask with DI water and add enough DI to flush the remaining sample through the separation funnel and drain onto the filter paper. Turn on vacuum to drain liquid. Repeat this process to ensure recovery of all of the LST, turning vacuum off between each rinse. Remove the filter funnel and place it in the 1000 ml drip flask. Empty vacuum flask into the dry down beaker with the rest of the LST that needs to be evaporated.
- viii. With a clean stirrer move the contents of the filter paper with the quartz in it from the funnel into a **labeled** Petri dish. Rinse the remaining grains from the funnel and the filter paper using a squirt bottle containing DI water. Put the used filter paper in the waste jar under the sink UNLESS you are saving minerals for other isotopic analyses including 36-Cl.
- x. Place the dish in the oven and dry overnight. When dry, transfer the contents of the Petri dish back into the appropriate 50 ml vial using a separate paper funnel for each sample. Note on the vial that it has been density separated and the date.
- xi. Store the vials in racks in the drawer labeled to 48 hour etch.
- ix. Rinse the vacuum flask, separation funnel, filter funnel in DI water in the acid hood and then return them to the density separation hood.
- x. Heavy liquid remaining from the mineral separation can be used again after the water has been evaporated and the liquid filtered to remove the mineral grains (as above). This evaporation is done on the hotplate at sub boiling temperatures. When the liquid has achieved a density at which the quartz chip floats, filter it back into storage bottle. Do not pour unfiltered liquid back in bottles. DO NOT LEAVE LIQUID UNCOVERED FOR LONG PERIODS OF TIME IN THE HOOD. IT GETS DIRTY...

If liquid remains cloudy after initial filtration, refilter with fine paper.

- xi. Bring up live batch in the database. Check off density separation on the database. The date will be automatically recorded.
- xii. Note: after extended use the heavy liquid will become dark, almost purple. It can be restored to its clear state with the addition of several drops of 30% hydrogen peroxide.
- e. Sample leaching in HF and HNO₃ (once for 48 hours)

Purpose: Final cleansing etch before purity test. **Processing time:** 48 hours.

Lab ware and materials needed:

Samples that have completed all prior steps. 4 liter PP Nalgene bottles. Labeling tape and sharpie. ultrasonic baths

Procedure:

- i. Label each of the one-liter Nalgene bottles (one for each of the samples).
- ii. Empty contents of sample vials into the bottles.
- iii. Fill bottles with DI water to within an inch or two from the top.
- iv. In the acid hood, dispense **25 ml of HF** (one 25 ml pumps) and **25 ml of HNO₃** into each Nalgene.
- v. Place 12 bottles in each of the ultrasounds. Top off water in baths (up to stains on the metal inserts).
- vi. Set timer for 24 hours.
- vii. Come in about 24 hours later and top off ultrasounds with water and reset timers to 24 hours.
- viii. After 48 hours, carefully decant and rinse each of the samples 4 to 5 times.
 - ix. Place bottles (without lids) in to the drying oven overnight.
 - x. When dry, weigh each sample on the digital balance and place them back into the appropriate 50 ml vials. Write on the vial that they have been 48 hour etched and the date.
- xi. Bring up live batch in the database. Check off 48 hour etch on the database. The weight and date will be automatically recorded. Store the vials in racks in the drawer labeled to purity testing.
- xii. Use ceramic magnet to clean quartz and remove any and all magnetic material by rotating tube next to magnet and drawing off magnetic material onto weigh paper.

Section 4 - Chemical ordering, waste disposal, and chemical storage

NOTE: It is always good to check the UIVM environmental safety page for updated information. See: <u>http://www.uvm.edu/~esf/wastedisposal/chemwaste.html</u> This website is a good place to start, especially the FAQ pages.

Ordering Chemicals

- 1. A tally of acid supplies is kept on the white board on the front of the oven. **Please keep this current.** The inventory should NEVER fall below 2 bottles of each acid.
- 2. When acid supplies run low in the mineral separation lab (2 bottles), replacement supplies should be ordered from chemsource. <u>http://www.uvm.edu/esf/chemsource</u>. A maximum of three bottles of each type of acid can be ordered. To order HCl use this link. An account must be set up using your UVM id and password and the chartstring for the lab to order online.
- 3. The lab has its own supply of HF and HNO₃ that is stored at ESF (Environmental Safety Facility). To order either of these chemicals call ESF at 65400.
- 4. LST is not available in the US, therefore it is important to note when supplies of this chemical are running low as it can take several weeks for new stock to arrive.

Acid Bottle Disposal

- 1. When acid bottles are empty, they need to be triple rinsed and have their labels removed or defaced. HCl and HNO₃ labels (glass bottles) have to be scraped off. HF (plastic bottles) labels will peel off.
- 2. A triple rinsed label must be attached to each triple rinsed bottle. The labels are stored in the top left hand drawer underneath the cork board.
- 3. 2.5 1 Glass bottles, once triple rinsed and labeled, go in the trash, they cannot be recycled.
- 4. 2.5 1 HF bottles, once triple rinsed and labeled, go in the trash.
- 5. If for some reason you use a small HF bottle (500 ml) with a pour spout, these cannot be rinsed and need to be tagged and disposed of as hazardous materials.

Storing Chemicals

1. All chemicals MUST be stored in secondary containment under fume hoods. When a new stock of chemicals arrive, they should be put away promptly in the appropriate secondary storage container under the fume hood. Whenever chemicals are unpacked, moved, or placed in storage full protective gear (rubber smock, goggles, and gloves) must be worn.

Collecting Chemical Waste and Waste Tags/Disposal

NOTE: Chemical waste containers must be labeled with a yellow waste accumulation label while you are collecting waste chemicals or chemical debris. **Be sure to write a date on the yellow label as soon as you begin collecting waste**. All chemicals should be listed on the label in full English (not chemical formulas).

- 1. Laboratory waste tags are located in the top left hand drawer underneath the cork board. A lab waste tag should be completed when waste containers such as those containing used filter paper are full.
- 2. Once the waste container is full and ready for disposal, fill out a Lab Waste Tag and attach it to the waste container. Enter the tag online at: <u>http://esf.uvm.edu/tags_entry/</u> Be sure to note where the waste is specifically located by noting such details in the "comments" section of the online tag. Entering the tag online lets Environmental Safety (ES) know that waste is ready for pickup in the lab. Legally, ES has 30 days to pick it up.
- 3. 1 liter round Nalgene bottles or 4 liter square plastic Nalgene bottles that are no longer useable because of age or cracking can be rinsed and recycled but they should be damaged before recycling to prevent others from removing them from the recycle bin and reusing (yes, it's happened before).

Acid Dumping Record Mineral Separation Lab (Rm 326)

		$\mathbf{A} = \mathbf{6N} \mathbf{H}$	ICI		
		$B = 1\% HNO_3 / 1\% HF$			
		C = 2% ł	HNO ₃ / 2	.5% HF	
					Neutralization
Date	Amount Dumped	C	ontent	s	System Response
		Α	В	С	
		Α	В	С	
		Α	В	С	
		Α	В	С	
		Α	В	С	
		Α	В	С	
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		Α	В	С	

Proje	Project Name:								Ultrasound:					
Processed By:														
Start	Date:													
#	Sample ID	2Х НС	CI Etch	Start Mass (g)	3X UI	trasoun Etch	d/HDR	Mass After 3X Ultrasound or HDR	Burn	Density Sep	Mass After Density Sen (g)	48 hr Etch	Mass After (g)	Notes:
		1st	2nd		1st	2nd	3rd	Etch (g)						
1														
2														
3														
4														
5														
6														
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Furnace Layout



Section 5 - Materials Safety Data Sheets

1. Nitric Acid

- 2. Hydrofloric Acid
- 3. Hydrocloric Acid
- 4. Hydrogen Peroxide

5. LST





Health3Fire0Reactivity0Personal
Protection

Material Safety Data Sheet Nitric acid, 65% MSDS

Section 1: Chemical Product and Company Identification					
Product Name: Nitric acid, 65%	Contact Information:				
Catalog Codes: SLN2161	Sciencelab.com, Inc. 14025 Smith Rd.				
CAS#: Mixture.	Houston, Texas 77396				
RTECS: Not applicable.	US Sales: 1-800-901-7247 International Sales: 1-281-441-4400				
TSCA: TSCA 8(b) inventory: Water; Nitric acid, fuming	Order Online: ScienceLab.com				
CI#: Not applicable.	CHEMTREC (24HR Emergency Telephone), call:				
Synonym: Nitric Acid, 65%	1-800-424-9300				
Chemical Name: Not applicable.	International CHEMTREC, call: 1-703-527-3887				
Chemical Formula: Not applicable.	For non-emergency assistance, call: 1-281-441-4400				

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Water	7732-18-5	35
Nitric acid, fuming	7697-37-2	65

Toxicological Data on Ingredients: Nitric acid, fuming: VAPOR (LC50): Acute: 244 ppm 0.5 hours [Rat]. 344 ppm 0.5 hours [Rat].

Section 3: Hazards Identification

Potential Acute Health Effects:

Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion, . Slightly hazardous in case of inhalation (lung sensitizer). Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Prolonged exposure may result in skin burns and ulcerations. Over-exposure by inhalation may cause respiratory irritation. Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.

Potential Chronic Health Effects:

CARCINOGENIC EFFECTS: Not available.

MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available.

The substance may be toxic to lungs, mucous membranes, upper respiratory tract, skin, eyes, teeth. Repeated or prolonged exposure to the substance can produce target organs damage. Repeated or prolonged contact with spray mist may produce chronic eye irritation and severe skin irritation. Repeated or prolonged exposure to spray mist may produce respiratory tract irritation leading to frequent attacks of bronchial infection.

Section 4: First Aid Measures

Eye Contact:

Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. Get medical attention immediately.

Skin Contact:

In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Cover the irritated skin with an emollient. Cold water may be used. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

Serious Skin Contact:

Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation:

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

Serious Inhalation:

Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek immediate medical attention.

Ingestion:

If swallowed, do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention immediately.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Non-flammable.

Auto-Ignition Temperature: Not applicable.

Flash Points: Not applicable.

Flammable Limits: Not applicable.

Products of Combustion: Not available.

Fire Hazards in Presence of Various Substances: of combustible materials

Explosion Hazards in Presence of Various Substances: Explosive in presence of reducing materials, of organic materials, of metals, of alkalis. Non-explosive in presence of open flames and sparks, of shocks.

Fire Fighting Media and Instructions: Not applicable.

Special Remarks on Fire Hazards:

Flammable in presence of cellulose or other combustible materials. Phosphine, hydrogen sulfide, selenide all ignite when fuming nitric acid is dripped into gas. (Nitric Acid, fuming)

Special Remarks on Explosion Hazards:

Reacts exlposively with metallic powders, carbides, cyanides, sulfides, alkalies and turpentine. Can react explosively with many reducing agents. Arsine, phosphine, tetraborane all oxidized explosively in presence of nitric acid. Cesium and rubidium acetylides explode in contact with nitric acid. Explosive reaction with Nitric Acid + Nitrobenzene + water. Detonation with Nitric Acid + 4-Methylcyclohexane. (Nitric acid, fuming)

Section 6: Accidental Release Measures

Small Spill:

Dilute with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container. If necessary: Neutralize the residue with a dilute solution of sodium carbonate.

Large Spill:

Corrosive liquid. Oxidizing material. Poisonous liquid.

Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not get water inside container. Avoid contact with a combustible material (wood, paper, oil, clothing...). Keep substance damp using water spray. Do not touch spilled material. Use water spray curtain to divert vapor drift. Use water spray to reduce vapors. Prevent entry into sewers, basements or confined areas; dike if needed. Call for assistance on disposal. Neutralize the residue with a dilute solution of sodium carbonate. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:

Keep locked up.. Keep container dry. Keep away from heat. Keep away from sources of ignition. Keep away from combustible material.. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Never add water to this product. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as reducing agents, combustible materials, organic materials, metals, acids, alkalis, moisture. May corrode metallic surfaces. Store in a metallic or coated fiberboard drum using a strong polyethylene inner package.

Storage:

Keep container tightly closed. Keep container in a cool, well-ventilated area. Separate from acids, alkalies, reducing agents and combustibles. See NFPA 43A, Code for the Storage of Liquid and Solid Oxidizers. Do not store above 23°C (73.4°F).

Section 8: Exposure Controls/Personal Protection

Engineering Controls:

Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:

Face shield. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves. Boots.

Personal Protection in Case of a Large Spill: Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:

TWA: 2 STEL: 4 (ppm) from ACGIH (TLV) [United States] TWA: 2 STEL: 4 from OSHA (PEL) [United States] Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties
Physical state and appearance: Liquid.
Odor: Acrid. Disagreeable and choking. (Strong.)
Taste: Not available.
Molecular Weight: Not applicable.
Color: Colorless to light yellow.
pH (1% soln/water): Acidic.
Boiling Point: 121°C (249.8°F)
Melting Point: -41.6°C (-42.9°F)
Critical Temperature: Not available.
Specific Gravity: 1.408 (Water = 1)
Vapor Pressure: 6 kPa (@ 20°C)
Vapor Density: 2.5 (Air = 1)
Volatility: Not available.
Odor Threshold: 0.29 ppm
Water/Oil Dist. Coeff.: Not available.
lonicity (in Water): Not available.
Dispersion Properties: See solubility in water, diethyl ether.
Solubility: Easily soluble in cold water, bot water,

Soluble in diethyl ether.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Incompatible materials

Incompatibility with various substances:

Highly reactive with alkalis. Reactive with reducing agents, combustible materials, organic materials, metals, acids.

Corrosivity:

Extremely corrosive in presence of aluminum, of copper. Non-corrosive in presence of glass, of stainless steel(304), of stainless steel(316), of brass.

Special Remarks on Reactivity:

A strong oxidizer. Reacts violently with alcohol, organic material, turpene, charcoal. Violent reaction with Nitric acid + Acetone and Sulfuric acid. Nitric Acid will react with water or steam to produce heat and toxic, corrosive and flammable vapors. (Nitric acid, fuming)

Special Remarks on Corrosivity:

In presence of traces of oxides, it attacks all base metals except aluminum and special chromium steels. It will attack some forms of plastics, rubber, and coatings. No corrosive effect on bronze. No corrosivity data for zinc, and steel

Polymerization: Will not occur.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation. Ingestion.

Toxicity to Animals:

LD50: Not available. LC50: Not available.

Chronic Effects on Humans:

Contains material which may cause damage to the following organs: lungs, mucous membranes, upper respiratory tract, skin, eyes, teeth.

Other Toxic Effects on Humans:

Extremely hazardous in case of inhalation (lung corrosive). Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (corrosive), of ingestion, .

Special Remarks on Toxicity to Animals: LDL - Lowest Published Lethal Dose [Human] - Route: Oral; Dose: 430 mg/kg (Nitric acid, fuming)

Special Remarks on Chronic Effects on Humans:

May cause adverse reproductive effects (effects on newborn and fetotoxicity) based on animal data. (Nitric acid, fuming)

Special Remarks on other Toxic Effects on Humans:

Acute Potential Health Effects:

Skin: Severely irritates skin. Causes skin burns and may cause deep and penetrating ulcers of the skin with a characteristic yellow to brownish discoloration. May be fatal if absorbed through skin.

Eyes: Severely irritates eyes. Causes eye burns. May cause irreversible eye injury.

Ingestion: May be fatal if swallowed. Causes serious gastrointestinal tract irritation or burns with nausea, vomiting, severe abdominal pain, and possible "coffee grounds" appearance of the vomitus . May cause perforation of the digestive tract.

Inhalation: May be fatal if inhaled. Vapor is extremely hazardous. Vapor may cause nitrous gas poisoning. Effects may be delayed. May cause irritation of the mucous membranes and respiratory tract with burning pain in the nose and throat, coughing, sneezing, wheezing, shortness of breath and pulmonary edema. Other symptoms may include nausea, and vomiting.

Chronic Potential Health Effects:

Repeated inhalation may produce changes in pulmonary function and/or chronic bronchitis. It may also affect behavior (headache, dizziness, drowsiness, muscle contaction or spasticity, weakness, loss of coordinaton, mental confusion), and urinary system (kidney faillure, decreased urinary output after several hours of

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:

Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are less toxic than the product itself.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

DOT Classification: Class 8: Corrosive material

Identification: : Nitric acid UNNA: 2031 PG: II

Special Provisions for Transport: Marine Pollutant

Section 15: Other Regulatory Information

Federal and State Regulations:

New York release reporting list: Nitric acid, fuming Rhode Island RTK hazardous substances: Nitric acid, fuming Pennsylvania RTK: Nitric acid, fuming Florida: Nitric acid, fuming Minnesota: Nitric acid, fuming Massachusetts RTK: Nitric acid, fuming New Jersey: Nitric acid, fuming TSCA 8(b) inventory: Water; Nitric acid, fuming SARA 302/304/311/312 extremely hazardous substances: Nitric acid, fuming SARA 313 toxic chemical notification and release reporting: Nitric acid, fuming 65% CERCLA: Hazardous substances.: Nitric acid, fuming: 1000 lbs. (453.6 kg);

Other Regulations: OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).

Other Classifications:

WHMIS (Canada):

CLASS D-1A: Material causing immediate and serious toxic effects (VERY TOXIC). CLASS D-2A: Material causing other toxic effects (VERY TOXIC). CLASS E: Corrosive liquid.

DSCL (EEC): R8- Contact with combustible material may cause fire. R35- Causes severe burns.

S23- Do not breathe gas/fumes/vapour/spray

S26- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.S36- Wear suitable protective clothing.S45- In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

HMIS (U.S.A.):

Health Hazard: 3

Fire Hazard: 0

Reactivity: 0

Personal Protection:

National Fire Protection Association (U.S.A.):

Health: 4

Flammability: 0

Reactivity: 0

Specific hazard:

Protective Equipment:

Gloves. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Face shield.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

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Last Updated: 10/10/2005 10:59 AM

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Health3Fire0Reactivity1Personal
Protection

Material Safety Data Sheet Hydrogen Peroxide 30% MSDS

Section 1: Chemical Product and Company Identification					
Product Name: Hydrogen Peroxide 30%	Contact Information:				
Catalog Codes: SLH1552	Sciencelab.com, Inc. 14025 Smith Rd.				
CAS#: Mixture.	Houston, Texas 77396				
RTECS: Not applicable.	US Sales: 1-800-901-7247 International Sales: 1-281-441-4400				
TSCA: TSCA 8(b) inventory: Water; Hydrogen Peroxide	Order Online: ScienceLab.com				
Cl#: Not applicable.	CHEMTREC (24HR Emergency Telephone), call:				
Synonym: Hydrogen Peroxide 30%	1-800-424-9300				
Chemical Name: Not applicable.	International CHEMTREC, call: 1-703-527-3887				
Chemical Formula: Not applicable.	For non-emergency assistance, call: 1-281-441-4400				

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Water	7732-18-5	70
Hydrogen Peroxide	7722-84-1	30

Toxicological Data on Ingredients: Hydrogen Peroxide: ORAL (LD50): Acute: 2000 mg/kg [Mouse]. DERMAL (LD50): Acute: 4060 mg/kg [Rat]. 2000 mg/kg [pig]. VAPOR (LC50): Acute: 2000 mg/m 4 hours [Rat].

Section 3: Hazards Identification

Potential Acute Health Effects:

Very hazardous in case of skin contact (irritant), of eye contact (irritant). Hazardous in case of skin contact (corrosive), of eye contact (corrosive), of ingestion, . Slightly hazardous in case of inhalation (lung sensitizer). Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Prolonged exposure may result in skin burns and ulcerations. Over-exposure by inhalation may cause respiratory irritation. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.

Potential Chronic Health Effects:

CARCINOGENIC EFFECTS: Not available.

MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance is toxic to lungs, mucous membranes. Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact:

Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. Get medical attention immediately.

Skin Contact:

In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Cover the irritated skin with an emollient. Cold water may be used. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

Serious Skin Contact:

Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation:

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

Serious Inhalation:

Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek immediate medical attention.

Ingestion:

Do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention if symptoms appear.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Non-flammable.

Auto-Ignition Temperature: Not applicable.

Flash Points: Not applicable.

Flammable Limits: Not applicable.

Products of Combustion: Not available.

Fire Hazards in Presence of Various Substances: combustible materials

Explosion Hazards in Presence of Various Substances: Slightly explosive in presence of open flames and sparks, of heat, of organic materials, of metals, of acids.

Fire Fighting Media and Instructions:

Fire: Small fires: Use water. Do not use dry chemicals or foams. CO2, or Halon may provide limited control. Large fires: Flood fire area with water from a distance. Move containers from fire area if you can do it without risk. Do not move cargo or vehicle if cargo has been exposed to heat. Fight fire from maximum distance or use unmanned hose holders or monitor nozzles. Cool containers with flooding quantities of water until well after fire is out. ALWAYS stay away from tanks engulfed in fire. For massive fire, use unmanned hose holders or monitor nozzles; if this is impossible, withdraw from area and let fire burn. /Hydrogen peroxide, aqueous solution, with not less than 8% but less than 20% Hydrogen peroxide; Hydrogen peroxide, aqueous solution, with not less than 20% but not more than 60% Hydrogen peroxide (stabilized as necessary)/ [QC Reviewed] [U.S. Department of Transportation. 2000 Emergency Response Guidebook. RSPA P 5800.8 Edition. Washington, D.C: U.S. Government Printing Office, 2000,p. G-140]

Special Remarks on Fire Hazards:

Nost cellulose (wood, cotton) materials contain enough catalyst to cause spontaneous ignition with 90% Hydrogen Peroxide. Hydrogen Peroxide is a strong oxider. It is not flammable itself, but it can cause spontaneous combustion of flammable materials and continued support of the combustion because it liberates oxygen as it decomposes.

Hydrogen peroxide mixed with magnesium and a trace of magnesium dioxide will ignite immediately.

Special Remarks on Explosion Hazards:

Soluble fuels (acetone, ethanol, glycerol) will detonate on a mixture with peroxide over 30% concentration, the violence increasing with concentration.

Explosive with acetic acid, acetic anhydride, acetone, alcohols, carboxylic acids, nitrogen containing bases, As2S3, Cl2 + KOH, FeS, FeSO4 + 2 methylpryidine + H2SO4, nitric acid, potassium permanganate, P2O5, H2Se, Alcohols + H2SO4, Alcohols + tin chloride, Antimoy trisulfide, chlorosulfonic acid, Aromatic hydrocarbons + trifluoroacetic acid, Azeliac acid + sulfuric acid (above 45 C), Benzenesulfonic anhydride, tert-butanol + sulfuric acid, Hydrazine, Sulfuric acid, Sodium iodate, Tetrahydrothiophene, Thiodiglycol, Mercurous oxide, mercuric oxide, Lead dioxide, Lead oxide, Manganese dioxide, Lead sulfide, Gallium + HCl, Ketenes + nitric acid, Iron (II) sulfate + 2-methylpyridine + sulfuric acid, Iron (II) sulfate + nitric acid, + sodium carboxymethylcellulose (when evaporated), Vinyl acetate, trioxane, water + oxygenated compounds (eg: acetaldehyde, acetic acid, acetone, ethanol, formaldehyde, formic acid, methanol, 2-propanol, propionaldehyde), organic compounds. Beware: Many mixitures of hydrogen peroxide and organic materials may not explode upon contact. However, the resulting combination is detonatable either upon catching fire or by impact.

EXPLOSION HAZARD: SEVERE, WHEN HIGHLY CONCENTRATED OR PURE H2O2 IS EXPOSED TO HEAT, MECHANICAL IMPACT, OR CAUSED TO DECOMPOSE CATALYTICALLY BY METALS & THEIR SALTS, DUSTS & ALKALIES.

ANOTHER SOURCE OF HYDROGEN PEROXIDE EXPLOSIONS IS FROM SEALING THE MATERIAL IN STRONG CONTAINERS. UNDER SUCH CONDITIONS EVEN GRADUAL DECOMPOSITION OF HYDROGEN PEROXIDE TO WATER + 1/2 OXYGEN CAN CAUSE LARGE PRESSURES TO BUILD UP IN THE CONTAINERS WHICH MAY BURST EXPLOSIVELY.

Fire or explosion:

May explode from friction, heat or contamination. These substances will accelerate burning when involved in a fire. May ignite combustibles (wood, paper, oil, clothing, etc.). Some will react explosively with hydrocarbons (fuels). Containers may explode when heated. Runoff may create fire or explosion hazard. /Hydrogen peroxide, aqueous solution, stabilized, with more than 60% Hydrogen peroxide; Hydrogen peroxide, stabilized/ [QC Reviewed] [U.S. Department of Transportation. 2000 Emergency Response Guidebook. RSPA P 5800.8 Edition. Washington, D.C: U.S. Government Printing Office, 2000,p. G-143].

Fire or explosion: These substances will accelerate burning when involved in a fire. Some may decompose explosively when heated or involved in a fire. May explode from heat or contamination. Some will react explosively with hydrocarbons (fuels). May ignite combustibles (wood, paper, oil, clothing, etc.). Containers may explode when heated. Runoff may create fire or explosion hazard. /Hydrogen peroxide, aqueous solution, with not less than 8% but less than 20% Hydrogen peroxide; Hydrogen peroxide, aqueous solution, with not less than 8% but less than 20% Hydrogen peroxide; Hydrogen peroxide, aqueous solution, with not less than 20% but not more than 60% Hydrogen peroxide (stabilized as necessary)/ [QC Reviewed] [U.S. Department of Transportation. 2000 Emergency Response Guidebook. RSPA P 5800.8 Edition. Washington, D.C: U.S. Government Printing Office, 2000,p. G-140]

(Hydrogen Peroxide)

Section 6: Accidental Release Measures

Small Spill:

Dilute with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container.

Large Spill:
Corrosive liquid. Oxidizing material.

Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not get water inside container. Avoid contact with a combustible material (wood, paper, oil, clothing...). Keep substance damp using water spray. Do not touch spilled material. Use water spray curtain to divert vapor drift. Prevent entry into sewers, basements or confined areas; dike if needed. Call for assistance on disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:

Keep locked up.. Keep container dry. Keep away from heat. Keep away from sources of ignition. Keep away from combustible material.. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Never add water to this product. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents, reducing agents, combustible materials, organic materials, metals, acids, alkalis.

Storage:

Keep container tightly closed. Keep container in a cool, well-ventilated area. Separate from acids, alkalies, reducing agents and combustibles. See NFPA 43A, Code for the Storage of Liquid and Solid Oxidizers. Do not store above 8°C (46.4°F). Refrigerate Sensitive to light. Store in light-resistant containers.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:

Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:

Face shield. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves. Boots.

Personal Protection in Case of a Large Spill:

Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:

Hydrogen Peroxide TWA: 1 (ppm) from ACGIH (TLV) [United States] TWA: 1 (ppm) from OSHA (PEL) [United States] TWA: 1 STEL: 2 [Canada] TWA: 1.4 (mg/m3) from NIOSH TWA: 1.4 (mg/m3) from OSHA (PEL) [United States] TWA: 1 (ppm) [United Kingdom (UK)] TWA: 1.4 (mg/m3) [United Kingdom (UK)]Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Odorless.

Taste: Slightly acid. Bitter

Molecular Weight: Not applicable.

Color: Clear Colorless.

pH (1% soln/water): Not available

Boiling Point: 108°C (226.4°F)

Melting Point: -33°C (-27.4°F)

Critical Temperature: Not available.

Specific Gravity: 1.1 (Water = 1)

Vapor Pressure: 3.1 kPa (@ 20°C)

Vapor Density: 1.1 (Air = 1)

Volatility: Not available.

Odor Threshold: Not available.

Water/Oil Dist. Coeff.: Not available.

lonicity (in Water): Not available.

Dispersion Properties: See solubility in water, diethyl ether.

Solubility: Easily soluble in cold water. Soluble in diethyl ether.

Section 10: Stability and Reactivity Data

Stability: The product is stable. It contains a stabilizer.

Instability Temperature: Not available.

Conditions of Instability: Excess heat, incompatible materials

Incompatibility with various substances: Reactive with reducing agents, combustible materials, organic materials, metals, acids, alkalis.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity:

Light sensitive.

Incompatible with reducing materials, ethers (dioxane, furfuran, tetrahydrofuran), oxidizing materials, Metals(eg. potassium, sodium lithium, iron, copper, brass, bronze, chromium, zinc, lead, silver, nickel), metal oxides (eg. cobalt oxide, iron oxide, lead oxide, lead hydroxide, manganese oxide), metal salts (eg. calcium permanganate, salts of iron), manganese, asbestos, vanadium, platinium, tungsten, molybdeum, triethylamine, palladium, sodium pyrophosphate, carboxylic acids, cyclopentadiene, formic acid, rust, ketones, sodium carbonate, alcohols, sodium borate, aniline, mercurous chloride, rust, nitric acid, sodium pyrophosphate, hexavalent chromium compounds, tetrahydrofuran, sodium fluoride organic matter, potassium permanganate, urea, chlorosulfonic acid, manganese dioxide, hydrogen selenide, charcoal, coal, sodium borate, alkalies, cyclopentadiene, glycerine, cyanides (potassium, cyanide, sodium cyanide), nitrogen compounds.

Caused to decompose catalytically by metals (in order of decreasing effectiveness): Osmium, Palladium, Platinum, Iridium, Gold, Silver, Manganese, Cobalt, Copper, Lead. Concentrated hydrogen peroxide may decompose violently or explosively in contact with iron, copper, chromium, and most other metals and their salts, and dust.

(Hydrogen Peroxide)

Special Remarks on Corrosivity: Not available.

Polymerization: Will not occur.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Eye contact.

Toxicity to Animals:

Acute oral toxicity (LD50): 6667 mg/kg (Mouse) (Calculated value for the mixture). Acute dermal toxicity (LD50): 6667 mg/kg (pig) (Calculated value for the mixture).

Chronic Effects on Humans:

CARCINOGENIC EFFECTS: Classified A3 (Proven for animal.) by ACGIH [Hydrogen Peroxide]. Classified 3 (Not classifiable for human.) by IARC [Hydrogen Peroxide].

MUTAGENIC EFFECTS: Mutagenic for mammalian somatic cells. [Hydrogen Peroxide]. Mutagenic for bacteria and/or yeast. [Hydrogen Peroxide].

Contains material which may cause damage to the following organs: blood, upper respiratory tract, skin, eyes, central nervous system (CNS).

Other Toxic Effects on Humans:

Very hazardous in case of skin contact (irritant).

Hazardous in case of skin contact (corrosive), of eye contact (corrosive), of ingestion, of inhalation (lung corrosive).

Special Remarks on Toxicity to Animals: Not available.

Special Remarks on Chronic Effects on Humans:

May cause cancer and may affect genetic material based on animal data. May be tumorigenic. (Hydrogen Peroxide)

Special Remarks on other Toxic Effects on Humans:

Acute Potential Health Effects:

Skin: Causes severe skin irritation and possible burns. Absorption into skin may affect behavior/central nervous system (tremor, ataxia, convulsions), respiration (dyspnea, pulmonary emboli), brain.

Eyes: Causes severe eye irritation, superficial clouding, corneal edema, and may cause burns.

Inhalation: Causes respiratory tract irritation with coughing, lacrimation. May cause chemical burns to the respiratory tract. May affect behavior/Central nervous system (insomnia, headache, ataxia, nervous tremors with numb extremities) and may cause ulceration of nasal tissue, and , chemical pneumonia, unconciousness, and possible death. At high concentrations, respiratory effects may include acute lung damage, and delayed pulmonary edema. May affect blood.

Ingestion: Causes gastrointestional tract irritation with nausea, vomiting, hypermotility, and diarrhea. Causes gastrointestional tract burns. May affect cardiovascular system and cause vascular collapse and damage. May affect blood (change in leukocyte count, pigmented or nucleated red blood cells). May cause difficulty in swallowing, stomach distension and possible cerebal swelling. May affect behavior/central nervous system (tetany, excitement).

Chronic Potential Health Effects:

Prolonged or repeated skin contact may cause dermatitis.

Repeated contact may also cause corneal damage.

Prolonged or repeated ingestion may affect metabolism (weight loss).

Prolonged or repeated inhalation may affect respiration, blood.

(Hydrogen Peroxide)

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation: Possibly hazardous short/long term degradation products are to be expected.

Toxicity of the Products of Biodegradation: The products of degradation are less toxic than the product itself.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

DOT Classification: CLASS 5.1: Oxidizing material.

Identification: : Hydrogen peroxide, aqueous solution UNNA: 2014 PG: II

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations:

New York acutely hazardous substances: Hydrogen Peroxide Rhode Island RTK hazardous substances: Hydrogen Peroxide Pennsylvania RTK: Hydrogen Peroxide Florida: Hydrogen Peroxide Minnesota: Hydrogen Peroxide Massachusetts RTK: Hydrogen Peroxide New Jersey: Hydrogen Peroxide TSCA 8(b) inventory: Hydrogen Peroxide SARA 302/304/311/312 extremely hazardous substances: Hydrogen Peroxide CERCLA: Hazardous substances.: Hydrogen Peroxide: 1 lbs. (0.4536 kg);

Other Regulations: OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).

Other Classifications:

WHMIS (Canada):

CLASS C: Oxidizing material. CLASS E: Corrosive liquid. CLASS F: Dangerously reactive material.

DSCL (EEC):

HMIS (U.S.A.):

Health Hazard: 3

Fire Hazard: 0

Reactivity: 1

Personal Protection:

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 0

Reactivity: 1

Specific hazard:

Protective Equipment:

Gloves. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Face shield.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

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Health3Fire0Reactivity0Personal
Protection

Material Safety Data Sheet Hydrofluoric Acid, 48% - 52% MSDS

Section 1: Chemical Product and Company Identification					
Product Name: Hydrofluoric Acid, 48% - 52% Contact Information:					
Catalog Codes: SLH2329, SLH3073	Sciencelab.com, Inc. 14025 Smith Rd.				
CAS#: 7664-39-3	Houston, Texas 77396				
RTECS: Not applicable.	US Sales: 1-800-901-7247 International Sales: 1-281-441-4400				
TSCA: TSCA 8(b) inventory: Water; Hydrofluoric acid	Order Online: ScienceLab.com				
CI#: Not available.	CHEMTREC (24HR Emergency Telephone), call:				
Synonym: Hydrogen Fluoride; Hydrofluoride	1-800-424-9300				
Chemical Name: Hydrofluoric acid	International CHEMTREC, call: 1-703-527-3887				
Chemical Formula: Not applicable.	For non-emergency assistance, call: 1-281-441-4400				

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight	
Water	7732-18-5	48-52	
Hydrogen fluoride	7664-39-3	48-52	

Toxicological Data on Ingredients: Hydrofluoric acid: VAPOR (LC50): Acute: 1276 ppm 1 hours [Rat]. 342 ppm 1 hours [Mouse]. 1774 ppm 1 hours [Monkey]. 4327 ppm 0.5 hours [Guinea pig].

Section 3: Hazards Identification

Potential Acute Health Effects:

Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion. Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, or, occasionally, blistering.

Potential Chronic Health Effects:

Non-corrosive for skin. Non-irritant for skin. Non-sensitizer for skin. Non-permeator by skin. Non-irritating to the eyes. Non-hazardous in case of ingestion. Non-hazardous in case of inhalation. Non-irritant for lungs. Non-sensitizer for lungs.

CARCINOGENIC EFFECTS: Not available. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available.

The substance may be toxic to lungs, mucous membranes, skin, eyes, bones, teeth.

Repeated or prolonged exposure to the substance can produce target organs damage. Repeated or prolonged contact with spray mist may produce chronic eye irritation and severe skin irritation. Repeated or prolonged exposure to spray mist may produce respiratory tract irritation leading to frequent attacks of bronchial infection. Repeated exposure to a highly toxic material may produce general deterioration of health by an accumulation in one or many human organs.

Section 4: First Aid Measures

Eye Contact:

Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15-30 minutes. Cold water may be used. Keep the eyelids apart and away from the eyeballs during irrigation. Do not use oily drops or ointment or HF skin burn treatments on the eyes. Get medical attention immediately, preferrably an eye specialist. If a physician is not immediately available, apply one or two drops of ophthalmic anesthetic (e.g. 0.5% Pontocaine Hydrochloride solution). Place ice pack on eyes until reaching emergency room.

Skin Contact:

In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Cover the irritated skin with an emollient. Cold water may be used. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

While waiting for medical attention, it has been shown that flushing the affected area with water for one minute and then massaging HF Antidote Gel into the wound until there is a cessation of pain is a most effective first aid treatment. HF Antidote Gel contains Calcium Gluconate which combines with HF for insoluble Calcium Fluoride, thus preventing the extraction of calcium from the body tissue and bones.

Another alternative first aid treatment, after thorough washing of the burned area, is to immerse the burned area in a solution of 0.2% iced aqueous Hyamine 1622 or 0.13% iced aqueous Zephiran Chloride. If immersion is inpractical, towels should be soaked with one of the above solutions and used as compresses for the burn area. Hyamine 1622 is a trade name for Tetracaine Benzethonium Chloride. Zephiran is a trade name for Benzalkonium Chloride.

Again, seek medical attention as soon as possible for all burns regardless of how minor they may appear initially.

Serious Skin Contact:

Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation:

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

Serious Inhalation:

Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek immediate medical attention.

Ingestion:

If swallowed, do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention immediately.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Non-flammable.

Auto-Ignition Temperature: Not applicable.

Flash Points: Not applicable.

Flammable Limits: Not applicable.

Products of Combustion: Not available.

Fire Hazards in Presence of Various Substances: Not applicable.

Explosion Hazards in Presence of Various Substances:

Explosive in presence of metals.

Non-explosive in presence of open flames and sparks, of shocks.

Fire Fighting Media and Instructions: Not applicable.

Special Remarks on Fire Hazards: Hazardous decompositon: May form acid vapors, hydrogen fluoride.

Special Remarks on Explosion Hazards:

It's corrosive action on metals can result in formation of hydrogen gas in containers and piping to create explosion hazard.

Reacts explosively with Cyanogen fluoride (polymerizes explosively), glycerol plus nitric acid (evolves gas from oxidation), methanesulfonic acid (evolves oxygen difluoride).

Hydrofluoric acid reacts with most metals to release hydrogen gas which can form explosive mixtures with air.

Section 6: Accidental Release Measures

Small Spill:

Dilute with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container. If necessary: Neutralize the residue with a dilute solution of sodium carbonate.

Large Spill:

Corrosive liquid. Poisonous liquid.

Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not get water inside container. Do not touch spilled material. Use water spray curtain to divert vapor drift. Use water spray to reduce vapors. Prevent entry into sewers, basements or confined areas; dike if needed. Call for assistance on disposal. Neutralize the residue with a dilute solution of sodium carbonate. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:

Do not ingest. Do not breathe gas/fumes/ vapor/spray. Never add water to this product. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as organic materials, metals, alkalis, moisture.

May corrode metallic surfaces and glass. Store in a polyethylene container.

Storage: Keep container tightly closed. Keep container in a cool, well-ventilated area.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:

Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:

Face shield. Synthetic (impervious) apron or full suit. A full impervious suit is recommended if exposure is possible to a large portion of the body. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves (impervious - neoprene, nitrile). Impervious Boots.

Personal Protection in Case of a Large Spill:

Splash goggles. Full suit (impervious). Vapor respirator. Impervious Boots. Gloves (impervious).. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:

Hydrogen fluoride STEL: 2.3 (mg/m3) from ACGIH (TLV) [United States] STEL: 3 (ppm) from ACGIH (TLV) [United States] CEIL: 6 from NIOSH CEIL: 5 (mg/m3) from NIOSH TWA: 3 STEL: 6 (ppm) from OSHA (PEL) [United States]Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Acrid (Strong.)

Taste: Not available.

Molecular Weight: Not applicable.

Color: Colorless. Clear

pH (1% soln/water): <2 [Acidic.]

Boiling Point: 108°C (226.4°F)

Melting Point: <-36.111°C (-33°F)

Critical Temperature: Not available.

Specific Gravity: 1.15 - 1.18 (Water = 1)

Vapor Pressure: 3.3 kPa (@ 20°C)

Vapor Density: 1.97 (Air = 1)

Volatility: Not available.

Odor Threshold: 0.5-3 ppm

Water/Oil Dist. Coeff.: Not available.

lonicity (in Water): Not available.

Dispersion Properties: See solubility in water, diethyl ether.

Solubility: Easily soluble in cold water, hot water. Partially soluble in diethyl ether.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Incompatible materials

Incompatibility with various substances:

Highly reactive with metals. Reactive with organic materials, alkalis.

Corrosivity:

Extremely corrosive in presence of glass, of aluminum, of stainless steel(304), of stainless steel(316). Slightly corrosive in presence of copper.

Special Remarks on Reactivity:

Incompatible with glass, ceramics, concrete, alkali materials, and will generate hydrogen gas on contact with metals, leather, rubber, common metals, carbonates, sulfides, cyanides, oxides of silicon, fluorine. Reacts violently with:

Acetic anhydride, 2-amino ethanol, Ammonium hydroxide, Arsenic trioxide, Bismuthic acid (produces oxygen), Calcium oxide, Chlorosulfonic acid, Dialuminum octavanadium tridecasilicide, Ethylene diamine, Ethyleneimine, Fluorine, Mercuric oxide, Mercury (II) oxide plus organic materials(above zero degree C), Nitric acid plus lactic acid (mixtures are unstable), Nitric acid plus propylene glycol, Olen-Phenylazopiperidine, Phosphoric anhydride (Phosphorus pentoxide unites with hydrogen fluoride vigorously, even at 19.5 degrees C, HSDB 1990), Potassium permanganate, Potassium tetrafluorosilicate(2-) (evolves silicon tetrafluoride gas), Propriolactone (beta-), Propylene glycol and silver nitrate (gas evolution and formation of silver fulminate), Propylene oxide, Sodium, Sodium hydroxide, Sodium tetrafluorosilicate, Sulfuric acid, Vinyl acetate.

Special Remarks on Corrosivity:

It corrodes most substances except lead, wax, polyethylene, and platinum. It will attack some forms of plastics, rubber and coatings. It attacks glass or stoneware, dissolving the silica. Minor corrosive effect on bronze.

Polymerization: Will not occur.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation. Ingestion.

Toxicity to Animals: Acute toxicity of the vapor (LC50): 342 1 hours [Mouse].

Chronic Effects on Humans: May cause damage to the following organs: lungs, mucous membranes, skin, eyes, bones, teeth.

Other Toxic Effects on Humans:

Extremely hazardous in case of inhalation (lung corrosive). Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (corrosive), of ingestion, .

Special Remarks on Toxicity to Animals: Not available.

Special Remarks on Chronic Effects on Humans:

May cause adverse reproductive effects (Fetotoxicity) based on animal data. May affect genetic material based on animal data. (Hydrogen fluoride)

Special Remarks on other Toxic Effects on Humans:

Acute Potential Health Effects:

Skin: Causes severe irritation and burns/irreversible destruction of skin. Readily penetrates skin and mucous membranes.

Eyes: Causes severe irritation and burns/irreversible destruction of eyes.

Inhalation: Causes severe irritation and burns/irreversible destruction of respiratory tract/lungs. May also affect behavior (change in motor activity, coma), blood, metabolism, sense organs, cardiovascular system (lowering of blood pressure, arrythmia), urinary system, gastrointestinal tract, respiration, and urinary system. Symptoms may

incluide severe throat irritation, cough, dyspnea, cyanosis, lung injury, and noncardiogenic pulmonary edema. Acute inhalation also depletes calcium levels in the body when can lead to hypocalcemia. Inhalation exposure of 50 ppm for 5 min. may be fatal.

Ingestion: Causes severe irritation and burns/irreversible destruction of digestive tract/stomach. Serious gastrointestinal effects may include hematemesis, nausea, and severe abdominal pain, painful necrotic leisons, hemorrhagic gastris, pancreatitis, local caustic effects to mouth and gastrointestinal tract. Severe systemic toxicity including hypocalcemia, hypomagnesemia, hyperkalemia, ventricular dysrhythmia and death may also occur.

Chronic Potential Health Effects: Repeated exposure to airborne concentrations of 3 ppm or less could be tolerated with no apparent ill effects for 6 hours/day for up to 50 days; redness of the skin and irritation and burning of the eyes and nose were noted at airborne concentrations between 3 ppm and 4.7 ppm (ACGIH, 1992). No significant changes in pulmonary function occurred with occupational exposure to airborne levels averaging 1.03 ppm (ACGIH).

Effects of chronic exposure by inhalation and ingestion include systemic fluoride toxicity (FLUOROSIS), skeletal/bone structure abnormalties (osteosclerosis, and mottling of the teeth (Clayton & Clayton, 1994; White, 1980; Waldbott & Lee, 1978). Hypocalcemia), metabolic acidosis, chronic bronchitis, pulmonary edema, and death can occur from high-level chronic exposure.

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:

Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are less toxic than the product itself.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

DOT Classification:

CLASS 6.1: Poisonous material. Class 8: Corrosive material

Identification: : Hydrofluoric Acid solution (Hydrogen fluoride) UNNA: 1790 PG: II

Special Provisions for Transport: Not available

Section 15: Other Regulatory Information

Federal and State Regulations:

Connecticut hazardous material survey.: Listed as Hydrofluoric acid or Hydrogen fluoride Illinois toxic substances disclosure to employee act: Listed as Hydrofluoric acid or Hydrogen fluoride Illinois chemical safety act: Listed as Hydrofluroic or Hydrogen fluoride New York release reporting list: Listed as Hydrofluoric acid Rhode Island RTK hazardous substances: Listed as Hydrofluoric acid or Hydrogen fluoride Pennsylvania RTK: Listed as Hydrofluoric acid Minnesota: Hydrogen fluoride Massachusetts RTK: Listed as Hydrogen fluoride Massachusetts spill list: Listed as Hydrofluoric acid or Hydrogen fluoride New Jersey: Listed as Hydrofluoric acid or Hydrogen fluoride Louisiana RTK reporting list: Listed as Hydrogen fluoride Louisiana spill reporting: Listed as Hydrogen fluoride Califoria Director's List of Hazardous substances: Listed as Hydrofluoric acid or Hydrogen Fluoride TSCA 8(b) inventory: Water; Hydrofluoric acid TSCA 4(a) proposed test rules: Listed as Hydrogen fluoride SARA 302/304/311/312 extremely hazardous substances: Listed as Hydrofluoric acid or Hydrogen fluoride SARA 313 toxic chemical notification and release reporting: Hydrogen fluoride 48% CERCLA: Hazardous substances.: Listed as Hydrofluoric acid or Hydrogen fluoride; 100 lbs. (45.36 kg);

Other Regulations:

OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200). EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances. INTERNATIOINAL LISTS: China (National Inventory): Listed as Hydrofluoric acid; Japan (ENCS - National Inventory): Listed as Hydrogen Fluoride; Korea (KECI - National Inventory): Listed as Hydrofluoric acid; Philippines (PICCS - National Inventory): Listed as Hydrofluoric acid

Other Classifications:

WHMIS (Canada):

CLASS D-1B: Material causing immediate and serious toxic effects (TOXIC). CLASS E: Corrosive liquid.

DSCL (EEC):

R26/27/28- Very toxic by inhalation, in contact with skin and if swallowed. R35- Causes severe burns. S7/9- Keep container tightly closed and in a well-ventilated place. S26- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. S36/37- Wear suitable protective clothing and gloves. S45- In case of accident or if you feel unwell, seek medical advice immediately (show the HMIS (U.S.A.): Health Hazard: 3 Fire Hazard: 0 Reactivity: 0 **Personal Protection:**

National Fire Protection Association (U.S.A.):

Health: 4

Flammability: 0

Reactivity: 1

Specific hazard:

Protective Equipment:

Gloves. Synthetic apron. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Face shield.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

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Health3Fire0Reactivity1Personal
Protection-

Material Safety Data Sheet Hydrochloric acid, 37% MSDS

Section 1: Chemical Product and Company Identification Product Name: Hydrochloric acid, 37% **Contact Information:** Sciencelab.com, Inc. Catalog Codes: SLH1360, SLH2206, SLH3052 14025 Smith Rd. Houston, Texas 77396 CAS#: Mixture. US Sales: 1-800-901-7247 RTECS: MW4025000 International Sales: 1-281-441-4400 TSCA: TSCA 8(b) inventory: Hydrochloric acid Order Online: ScienceLab.com Cl#: Not applicable. CHEMTREC (24HR Emergency Telephone), call: 1-800-424-9300 **Synonym:** Hydrochloric Acid; Muriatic Acid; Hydrochloric Acid 37% N.F, F.C.C., Electronic/Cleanroom grades International CHEMTREC, call: 1-703-527-3887 Chemical Name: Not applicable. For non-emergency assistance, call: 1-281-441-4400 Chemical Formula: Not applicable.

Section 2: Composition and Information on Ingredients

composition.				
Name	CAS #	% by Weight		
Hydrogen chloride	7647-01-0	37		
Water	7732-18-5	63		

Toxicological Data on Ingredients: Hydrogen chloride: GAS (LC50): Acute: 4701 ppm 0.5 hours [Rat].

Section 3: Hazards Identification

Potential Acute Health Effects:

Composition

Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion, . Slightly hazardous in case of inhalation (lung sensitizer). Non-corrosive for lungs. Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.

Potential Chronic Health Effects:

Slightly hazardous in case of skin contact (sensitizer). CARCINOGENIC EFFECTS: Classified 3 (Not classifiable for human.) by IARC [Hydrochloric acid].

MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available.

The substance may be toxic to kidneys, liver, mucous membranes, upper respiratory tract, skin, eyes, Circulatory System, teeth.

Repeated or prolonged exposure to the substance can produce target organs damage. Repeated or prolonged contact with spray mist may produce chronic eye irritation and severe skin irritation. Repeated or prolonged exposure to spray mist may produce respiratory tract irritation leading to frequent attacks of bronchial infection. Repeated exposure to a highly toxic material may produce general deterioration of health by an accumulation in one or many human organs.

Section 4: First Aid Measures

Eye Contact:

Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. Get medical attention immediately.

Skin Contact:

In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Cover the irritated skin with an emollient. Cold water may be used. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

Serious Skin Contact:

Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation:

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.

Serious Inhalation:

Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek immediate medical attention.

Ingestion:

If swallowed, do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention immediately.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Non-flammable.

Auto-Ignition Temperature: Not applicable.

Flash Points: Not applicable.

Flammable Limits: Not applicable.

Products of Combustion: Not available.

Fire Hazards in Presence of Various Substances: of metals

Explosion Hazards in Presence of Various Substances: Non-explosive in presence of open flames and sparks, of shocks.

Fire Fighting Media and Instructions: Not applicable.

Special Remarks on Fire Hazards:

Non combustible.

Calcium carbide reacts with hydrogen chloride gas with incandescence.

Uranium phosphide reacts with hydrochloric acid to release spontaneously flammable phosphine.

Rubidium acetylene carbides burns with slightly warm hydrochloric acid.

Lithium silicide in contact with hydrogen chloride becomes incandescent. When dilute hydrochloric acid is used, gas spontaneously flammable in air is evolved.

Magnesium boride treated with concentrated hydrochloric acid produces spontaneously flammble gas.

Cesium acetylene carbide burns hydrogen chloride gas.

Cesium carbide ignites in contact with hydrochloric acid unless acid is dilute.

Reacts with most metals to produce flammable Hydrodgen gas.

Special Remarks on Explosion Hazards:

Hydrogen chloride in contact with the following can cause an explosion, ignition on contact, or other violent/vigorous reaction: Acetic anhydride AgCIO + CCl4 Alcohols + hydrogen cyanide, Aluminum Aluminum-titanium alloys (with HCl vapor), 2-Amino ethanol, Ammonium hydroxide, Calcium carbide Ca3P2 Chlorine + dinitroanilines (evolves gas), Chlorosulfonic acid Cesium carbide Cesium acetylene carbide, 1,1-Difluoroethylene Ethylene diamine Ethylene imine, Fluorine, HClO4 Hexalithium disilicide H2SO4 Metal acetylides or carbides, Magnesium boride, Mercuric sulfate, Oleum, Potassium permanganate, beta-Propiolactone Propylene oxide Rubidium carbide, Rubidium, acetylene carbide Sodium (with aqueous HCl), Sodium hydroxide Sodium tetraselenium, Sulfonic acid, Tetraselenium tetranitride, U3P4, Vinyl acetate. Silver perchlorate with carbon tetrachloride in the presence of hydrochloric acid produces trichloromethyl perchlorate which detonates at 40 deg. C.

Section 6: Accidental Release Measures

Small Spill:

Dilute with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container. If necessary: Neutralize the residue with a dilute solution of sodium carbonate.

Large Spill:

Corrosive liquid. Poisonous liquid.

Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not get water inside container. Do not touch spilled material. Use water spray curtain to divert vapor drift. Use water spray to reduce vapors. Prevent entry into sewers, basements or confined areas; dike if needed. Call for assistance on disposal. Neutralize the residue with a dilute solution of sodium carbonate. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:

Keep locked up.. Keep container dry. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Never add water to this product. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents, organic materials, metals, alkalis, moisture. May corrode metallic surfaces. Store in a metallic or coated fiberboard drum using a strong polyethylene inner package.

Storage: Keep container tightly closed. Keep container in a cool, well-ventilated area.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:

Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:

Face shield. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves. Boots.

Personal Protection in Case of a Large Spill:

Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:

CEIL: 5 (ppm) from OSHA (PEL) [United States] CEIL: 7 (mg/m3) from OSHA (PEL) [United States] CEIL: 5 from NIOSH CEIL: 7 (mg/m3) from NIOSH TWA: 1 STEL: 5 (ppm) [United Kingdom (UK)] TWA: 2 STEL: 8 (mg/m3) [United Kingdom (UK)]Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Pungent. Irritating (Strong.)

Taste: Not available.

Molecular Weight: Not applicable.

Color: Colorless to light yellow.

pH (1% soln/water): Acidic.

Boiling Point: 50.5°C (122.9°F)

Melting Point: -46.2°C (-51.2°F) to -25.4 C

Critical Temperature: Not available.

Specific Gravity: 1.19 (Water = 1)

Vapor Pressure: 16 kPa (@ 20°C) average

Vapor Density: 1.267 (Air = 1)

Volatility: Not available.

Odor Threshold: 0.25 to 10 ppm

Water/Oil Dist. Coeff.: Not available.

lonicity (in Water): Not available.

Dispersion Properties: See solubility in water, diethyl ether.

Solubility: Soluble in cold water, hot water, diethyl ether.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Incompatible materials, water

Incompatibility with various substances:

Highly reactive with metals. Reactive with oxidizing agents, organic materials, alkalis, water.

Corrosivity:

Extremely corrosive in presence of aluminum, of copper, of stainless steel(304), of stainless steel(316). Non-corrosive in presence of glass.

Special Remarks on Reactivity:

Reacts with water especially when water is added to the product.

Absorption of gaseous hydrogen chloride on mercuric sulfate becomes violent @ 125 deg. C.

Sodium reacts very violently with gaseous hydrogen chloride.

Calcium phosphide and hydrochloric acid undergo very energetic reaction.

It reacts with oxidizers releasing chlorine gas.

Incompatible with, alkali metals, carbides, borides, metal oxides, vinyl acetate, acetylides, sulphides, phosphides, cyanides, carbonates.

Reacts with most metals to produce flammable Hydrogen gas.

Reacts violently (moderate reaction with heat of evolution) with water especially when water is added to the product. Isolate hydrogen chloride from heat, direct, alkalies (reacts vigorously), organic materials, and oxidizers (especially nitric acid and chlorates), amines, metals, copper and alloys (e.g. brass), hydroxides, zinc (galvanized materials), lithium silicide (incandescence), sulfuric acid(increase in temperature and pressure)

Hydrogen chloride gas is emitted when this product is in contact with sulfuric acid.

Adsorption of Hydrochloric Acid onto silicon dioxide results in exothmeric reaction.

Hydrogen chloride causes aldehydes and epoxides to violently polymerize.

Hydrogen chloride or Hydrochloric Acid in contact with the folloiwing can cause explosion or ignition on contact or other violent/vigorous reaction: Acetic anhydride, Alcohols + hydrogen cyanide, Aluminum, Aluminum phosphide, Aluminum-titanium alloys (with HCl vapor), 2-Amino ethanol, Ammonium, Ammonium hydroxide, 1,4-Benzoquinone diimine, Calcium acetylide (incandescence upon warming), Calcium carbide, Calcium phosphide, Carbon tetrachloride + silver perchlorate (produce trichlormethyl perchlorate), Cesium acetylene carbide, Cesium carbide, Cesium telluroacylates, Chlorine + dinitroanilines (evolves gas), Chloroacetaldehyde oxime, Chlorosulfonic acid, Cyanogen chloride (when catalyzed by HCl), 1,1-Difluoroethylene, Dinitroanilines, Ethylene, Ethylene diamine, Ethyl 2-formylpropionate oxime (when generated by using HCl as a catalyst),

Special Remarks on Corrosivity:

Highly corrosive. Incompatible with copper and copper alloys. It attacks nearly all metals (mercury, gold, platinium, tantalum, silver, and certain alloys are exceptions).

It is one of the most corrosive of the nonoxidizing acids in contact with copper alloys.

No corrosivity data on zinc, steel.

Severe Corrosive effect on brass and bronze

Polymerization: Will not occur.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation.

Toxicity to Animals:

Acute oral toxicity (LD50): 900 mg/kg [Rabbit]. Acute toxicity of the vapor (LC50): 1108 ppm, 1 hours [Mouse]. Acute toxicity of the vapor (LC50): 3124 ppm, 1 hours [Rat]. (Hydrochloric Acid)

Chronic Effects on Humans:

CARCINOGENIC EFFECTS: Classified 3 (Not classifiable for human.) by IARC [Hydrochloric acid]. May cause damage to the following organs: kidneys, liver, mucous membranes, upper respiratory tract, skin, eyes, Circulatory System, teeth.

Other Toxic Effects on Humans:

Very hazardous in case of skin contact (corrosive, irritant, permeator), of ingestion, . Hazardous in case of eye contact (corrosive), of inhalation (lung corrosive).

Special Remarks on Toxicity to Animals:

Lowest Published Lethal Doses (LDL/LCL) LDL [Man] -Route: Oral; 2857 ug/kg LCL [Human] - Route: Inhalation; Dose: 1300 ppm/30M LCL [Rabbit] - Route: Inhalation; Dose: 4413 ppm/30M

Special Remarks on Chronic Effects on Humans:

May cause adverse reproductive effects (fetoxicity). May affect genetic material.

Special Remarks on other Toxic Effects on Humans:

Acute Potential Health Effects:

Skin: Corrosive. Causes severe skin irritation and burns.

Eyes: Corrosive. Causes severe eye irritation/conjuntivitis, burns, corneal necrosis.

Inhalation: May be fatal if inhaled. Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract. Inhalation of hydrochloric acid fumes produces nose, throat, and larryngeal burning, and irritation, pain and inflammation, coughing, sneezing, choking sensation, hoarseness, laryngeal spasms, upper respiratory tract edema, chest pains, as well has headache, and palpitations. Inhalation of high concentrations can result in corrosive burns, necrosis of bronchial epithelium, constriction of the larynx and bronchi, nasospetal perforation, glottal closure,

occur, particularly if exposure is prolonged. May affect the liver.

Ingestion: May be fatal if swallowed. Causes irritation and burning, ulceration, or perforation of the gastrointestinal tract and resultant peritonitis, gastric hemorrhage and infection. Can also cause nausea, vomitting (with "coffee ground" emesis), diarrhea, thirst, difficulty swallowing, salivation, chills, fever, uneasiness, shock, strictures and stenosis (esophogeal, gastric, pyloric). May affect behavior (excitement), the cardiovascular system (weak rapid pulse, tachycardia), respiration (shallow respiration), and urinary system (kidneys- renal failure, nephritis).

Acute exposure via inhalation or ingestion can also cause erosion of tooth enamel.

Chronic Potential Health Effects:

Prolonged or repeated inhalation and/or ingestion may affect liver, bleeding of nose and gums, nasal and oral mucosal ulceration, conjunctivitis, respiratory tract (changes in pulmonary function, chronic bronchitis, overt respiratory tract abnormalities), teeth (yellowing of teethand erosion of tooth enamel), kidneys, and behavior (muscle contraction or spasticity).

Prolonged or repeated skin contact may cause dermatitis.

Prolonged or repeated eye contact with vapor/mist can cause conjunctivitis

dyspnea, bronchitis. Chemical pneumonitis and pulmonary edema can also

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:

Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are less toxic than the product itself.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

DOT Classification: Class 8: Corrosive material

Identification: : Hydrochloric acid, solution UNNA: 1789 PG: II

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations:

Connecticut hazardous material survey .: Hydrochloric acid Illinois toxic substances disclosure to employee act: Hydrochloric acid Illinois chemical safety act: Hydrochloric acid New York release reporting list: Hydrochloric acid Rhode Island RTK hazardous substances: Hydrochloric acid Pennsylvania RTK: Hydrochloric acid Minnesota: Hydrochloric acid Massachusetts RTK: Hydrochloric acid Massachusetts spill list: Hydrochloric acid New Jersey: Hydrochloric acid New Jersey spill list: Hydrochloric acid Louisiana RTK reporting list: Hydrochloric acid Louisiana spill reporting: Hydrochloric acid California Director's List of Hazardous Substances: Hydrochloric acid TSCA 8(b) inventory: Hydrochloric acid TSCA 4(a) proposed test rules: Hydrochloric acid SARA 302/304/311/312 extremely hazardous substances: Hydrochloric acid SARA 313 toxic chemical notification and release reporting: Hydrochloric acid CERCLA: Hazardous substances.: Hydrochloric acid: 5000 lbs. (2268 kg)

Other Regulations:

OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200). EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

Other Classifications:

WHMIS (Canada): CLASS D-2A: Material causing other toxic effects (VERY TOXIC). CLASS E: Corrosive liquid.

DSCL (EEC):

R25- Toxic if swallowed.
R26- Very toxic by inhalation.
R34- Causes burns.
S1/2- Keep locked up and out of the reach of children.
S26- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
S28- After contact with skin, wash immediately with plenty of [***]
S36/37/39- Wear suitable protective clothing, gloves and eye/face protection.
S45- In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

HMIS (U.S.A.):

Health Hazard: 3

Fire Hazard: 0

Reactivity: 1

Personal Protection:

National Fire Protection Association (U.S.A.):

Health: 3

Flammability: 0

Reactivity: 1

Specific hazard:

Protective Equipment:

Gloves. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Face shield.

Section 16: Other Information

References:

-Hawley, G.G.. The Condensed Chemical Dictionary, 11e ed., New York N.Y., Van Nostrand Reinold, 1987. -SAX, N.I. Dangerous Properties of Indutrial Materials. Toronto, Van Nostrand Reinold, 6e ed. 1984. -The Sigma-Aldrich Library of Chemical Safety Data, Edition II. -Guide de la loi et du règlement sur le transport des marchandises dangeureuses au canada. Centre de conformité internatinal Ltée. 1986.

Other Special Considerations: Not available.

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LST HEAVY LIQUID

Page 1 of 4

Date of issue: 15 April 2003

Use:

MATERIAL SAFETY DATA SHEET

Hazardous according to criteria of Worksafe Australia.

	COMPANY DETAILS			
COMPANY: ABN:	Central Chemical Consulting Pty Ltd 21 009 431 494			
ADDRESS:	PO Box 2546, Malaga, Western Australia 6944			
INFORMATION TELEPHONE NUMBER:	61 8 9248 2739			
EMERGENCY TELEPHONE NO:	61 8 9248 2739 <i>or</i> 61 8 9246 1102 (out of hours)			
FACSIMILE NUMBER:	61 8 9248 2749			

IDENTIFICATION					
Product Name: LST Heavy Liquid.					
Description:	Solution of lithium heteropolytungstates in water.				
Manufacturers Code:	Not applicable.				
UN Number:	No UN Number allocated.				
Dangerous Goods Class and Subsidiary Risk:	Not a dangerous good.				
Hazchem Code:	None allocated.				
Poisons Schedule:	Not scheduled.				

Heavy liquid for density separation of minerals and ores.

Physical Description/Properties:

Appearance:	Clear to pale coloured liquid.			
Melting Point:	Not Applicable.			
Vapour Pressure:	Negligible vapour pressure of solids.			
Specific Gravity:	2 850 kg/m ³ .			
Flash Point:	Not flammable.			
Flammability Limits:	Not flammable.			
Solubility Limits:	Limits: Miscible with water.			
Ingredients:				
NAME	CAS NO	Proportion		
Lithium heteropolytungstates	-	>80%		

HEALTH HAZARD INFORMATION

HEALTH EFFECTS

ACUTE EFFECTS:

Swallowed:	Unlikely to cause significant effects. Not expected to a route of exposure.			
Eye:	Crystalline solids are a severe eye irritant. May cause irreversible damage.			
Skin:	Non-irritant.			
Inhaled:	Not likely to be a route of exposure.			
CHRONIC EFFECTS:				
	Repeated dose studies in animals suggest irritation to gastrointestinal tract may occur.			

LST HEAVY LIQUID

FIRST AID:

Swallowed:	If swallowed and feeling unwell contact Doctor or Poisons Information Centre.		
Eye:	If in eye rinse out with running water for at least 15 minutes. Contact a doctor immediately.		
Skin:	If on skin wash off immediately with water.		
Advice to Doctor:	Treat Symptomatically.		

PRECAUTIONS FOR USE					
Exposure Limits:	No exposure standards have been established for this substance by Worksafe Australia.				
Engineering Controls:	The substance should be used in systems which recover and recycle LST Heavy Liquid.				
Personal Protection:					
Skin:	Impermeable rubber, vinyl or nitrile gloves should be worn when handling the material.				
Eyes:	Safety glasses/goggles must be worn when handling LST Heavy Liquid or its solids.				
Respiratory:	Not likely to be a route of exposure. Immediately clean up spills to prevent accumulation of dust from dried LST Heavy Liquid.				
Ingestion:	Do not allow eating, drinking or smoking in areas where LST Heavy Liquid is to be stored or used.				
Flammability:	Not flammable.				

LST HEAVY LIQUID

Page 4 of 4

SAFE HANDLING INFORMATION				
Storage and Transport:	The material is not a dangerous good. The material may have slight oxidising characteristics. Store in plastic containers out of direct sunlight.			
Spills and Disposal:	If spilled do not allow substance to enter waterways. If liquid, dike area and absorb with inert material such as sawdust. Dispose of in accordance with Federal, State and Local requirements.			
Fire/Explosion Hazard:	Will not present fire/explosion hazard.			

OTHER INFORMATION ON CRYSTALLINE LST SOLIDS:

Acute Oral LD 50 (rat) > 2 000 mg/kg Acute dermal (rat) > 2 000 mg/kg Non-irritant to skin (Rabbit) Irreversible damage to eye (crystals under Rabbit eyelid) Non skin-sensitiser (Guinea Pig) Non mutagenic in Salmonella typhimurium Not clastogenic in Mouse Micronucleus Assay LC50 (96 hr) in Fathead Minnow 94.7 mg/L LC50 (48 hr) Daphnia magna 288.6 mg/L LC50 (14 day) Daphnia magna 2.47 mg/L

CONTACT POINT:

Technical Manager 61 8 9248 2739

Section 6 – Operating Manuals

- 1, Ultrasonicators
- 2. Acid Dispensers
 - 3. Drying Oven
 - 4. Furnace



Ultrasonic Cleaners Models 1510, 2510, 3510, 5510, 8510



M

Operator's Manual www.Bransonic.com

CPN-214-142 Rev. B

Ultrasonic Cleaners Models 1510, 2510, 3510, 5510, 8510

PROCESS EQUIPMENT & SUPPLY, INC. 31255 Lorain Road Cleveland, Ohio 44070 1-800-539-6336 • Fax 1-800-539-6371 WWW.PROEQUIP.COM

Warranty

Ultrasonic Cleaners, when used in accordance with manufacturer's instructions and under normal use, are guaranteed for **two years after date of shipment.** Within the period guaranteed, Branson Ultrasonics Corporation will repair or replace free of charge, at its sole discretion, all parts that are defective because of material or workmanship, not including costs for removing or installing parts.

The Manufacturer's liability, whether based on warranty, negligence or other cause, arising out of and/or incidental to sale, use or operation of the transducer elements, or any part thereof, shall not in any case exceed the cost of repair or replacement of the defective equipment, and such repair or replacement shall be the exclusive remedy of the purchaser, and in no case shall the manufacturer be responsible for any and/or all consequential or incidental damages including without limitation, and/or all consequential damages arising out of commercial losses.

- Do not place parts or containers directly on the bottom of the cleaning tank; use a tray or wire to suspend items.
- Do not allow the solution to drop more than 3/8 inch below the operating level line with the cleaner on.
- Do not ever use alcohol, gasoline or flammable solutions. Doing so could cause a fire or explosion. Use only water-based solutions.
- Do not use mineral acids. These could damage the tank.

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7 70 10 14 16 18 19 21	Operating Your Cleaner Operating Your MT or MTH Cleaner Operating Your DTH Cleaner Draining Your Cleaner Optimizing Your Cleaner Application Hints Cleaning Methods Cleaning Solutions			
24	Troubleshooting			
28	Service Centers			



✓ Safety Precautions ▲

Before using your Ultrasonic Cleaner, please read and thoroughly understand these safety precautions. Failure to follow them may result in serious personal injury or property damage.

To avoid electrical shock:

- Do unplug from power source before filling or emptying the tank.
- Do keep the control panel and the area around the cleaner clean and dry -- wipe up solution which spills over the tank brim. Water and high voltage can cause electrical shock.
- Do not operate the cleaner without proper grounding.
- Do not remove the grounding prong on the line cord plug.
- Do not disassemble your cleaner -- high voltage inside the cleaner is dangerous.
- Do not immerse the cleaner in water.

To prevent personal and/or property damage:

- Do operate the cleaner with a vented cover or no cover.
- Do use water-based solutions.
- Do not ever use alcohol, gasoline or flammable solutions. Doing so could cause a fire or explosion and will void your warranty. Use only water-based solutions.
- Do not ever use mineral acids. These could damage the tank.
- Do not touch the stainless steel tank or cleaning solution -- they may be hot.
- Do not allow fluid temperature to exceed 70°C (160°F).
- Do not place your fingers or hands into the tank while the cleaner is operating. Doing so may cause discomfort and possible skin irritation. Avoid contact with solutions and provide adequate ventilation.
- Do not use solutions containing chlorine bleach.

To prevent damage to the cleaner:

- Do change your solution regularly.
- Do not cover vents on the cover.
- Do not operate the cleaner dry.
- Do not place parts or containers directly on the bottom of the cleaning tank; use a tray or wire to suspend items. Failure to comply may cause transducer damage and will void your warranty.
- Do not allow the solution to drop more than 3/8 inch below the operating level line with heat or ultrasonics on. Failure to comply may cause transducer and/or heater damage and will void your warranty.

Introduction

Ultrasonic Cleaners

This line of ultrasonic cleaners include five models with sizes ranging from 1/2 gallons, 3/4 gallons, 1-1/2 gallons, 2-1/2 gallons and 5-1/2 gallons. Each model is constructed using durable industrial style 40kHz transducers. These provide increased cleaning power along with built in sweep frequency to ensure uniform cleaning activity throughout the bath. The 1/2 and 3/4 gallon models have a molded dip in their rims to facilitate emptying of solution from the tank. The three larger sizes have built in drains and are supplied with tank drain kits. Each model can be purchased in three different configurations -- with a Mechanical Timer (MT), with a Mechanical Timer plus Heat (MTH) and with Digital Control, plus Heat and Timer (DTH).



When you first fill your unit, or refill it with fresh solution, use warm water for the solution. Turn on the heater (press the HEAT switch, if available), turn on the ultrasonics (press SONICS or rotate the Timer), add the cover and the solution will heat quickly to temperature.

Accessories For Your Cleaner

Optional accessories include regular and beaker positioning covers, solid and perforated insert trays, mesh baskets and beakers.

Unpacking Your Cleaner

Please check your cleaner and its carton carefully for any external or internal damage. **If you find damage, contact your shipping carrier immediately,** before contacting your distributor. Please retain your packaging for future use.

Installing Your Cleaner

Check the plate on the back of the cleaner for correct power requirements. Position your cleaner within easy reach of a standard grounded electrical outlet. Do not place the cleaner on a circuit which could become overloaded.

If your cleaner does not operate correctly, first refer to the troubleshooting section for possible causes, or contact an authorized service center listed at the back of this manual, for additional information.

Equipment Specifications

Tank Capacity	Tank Size	Overall Size	Weight	Max Input Power	Heater Power	Max. Draw Power Req. (Watts)*
1/2 gal. (1.91 L)	L: 6" W: 5.5" D: 4"	L: 10" W: 12" D: 11.5"	7 lbs. (3.2KG)	80W	0 63 63	80 143 143
3/4 gal. (2.81 L)	L: 9.5" W: 5.5" D: 4"	L: 13.5" W: 12" D: 11.5"	9 lbs. (4. KG)	130W	0 109 109	130 239 239
1-1/2 gal. (5.71 L)	L: 11.5" W: 6" D: 6"	L: 16" W: 12" D: 14.5"	12 lbs. (5.4 KG)	130W	0 205 205	130 335 335
2-1/2 gal. (9.51 L)	L: 11.5" W: 9.5" D: 6"	L: 16" W: 15.5" D: 14.5"	14 lbs. (6.4 KG)	185W	0 284 284	185 469 469
5-1/2 gal. (20.81 L)	L: 19.5" W:11.5" D: 6"	L: 24" W: 18" D: 14.5"	26 lbs. (11.8 KG)	320W	0 561 561	320 881 881

NOTE:

- All models have a frequency of 40kHz.
- In DTH cleaners, the temperature readout accuracy is ± 4°C.
- $120V \pm 10\%$, 60Hz is optimum voltage for all cleaners.
- All cleaners have CSA approval and comply with FCC regulations.
- All 220V units meet CE standards.
- * indicates power levels for MT, MTH and DTH cleaners, in that order.
- Units will cause GFI protected outlets to trip.
- All units have a ground leakage current less than .50ma.
How Ultrasonics Cleaning Works

Ultrasonic sound is sound transmitted at frequencies generally beyond the range of human hearing. In your ultrasonic cleaner, ultrasonic sound (sonics) is used for cleaning materials and parts. This is how it works:



As the sound waves from the transducer radiate through the solution in the tank, they cause alternating high and low pressures in the solution.



During the low pressure stage, millions of microscopic bubbles form and grow. This process is called CAVITATION, meaning "formation of cavities".



During the high pressure stage, the bubbles collapse, or "implode" releasing enormous amounts of energy. These implosions act like an army of tiny scrub brushes. They work in all directions, attacking every surface and invading all recesses and openings.

6 Introduction

Operating Your Cleaner

If this is the first time you are using the cleaner, please read this whole section before operating your cleaner.

Operating Your MT or MTH Cleaner



MT Cleaner

MTH Cleaner



Explanation of Controls

Control	Function	
HEAT	Activates heat to 60°C maximum.	
(MTH only)	NOTE: Refer to pages 16 and 17 for further	
	temperature information.	
TIMER	Activates ultrasonics and sets time.	
	Use to turn unit Off.	
	Turn clockwise for variable time 0-60 mins. Turn counterclockwise to hold position for continuous operation.	

7 0 11 11 01

Before You Start Cleaning

- Do not place parts or containers directly on the bottom of the cleaning tanks; use a tray or wire to suspend items.
- Do not allow the solution to drop more than 3/8 inch below the operating level line with the cleaner on.
- Do not ever use alcohol, gasoline or flammable solutions. Doing so could cause a fire or explosion. Use only water-based solutions.
- Do not ever use mineral acids. These could damage the tank.

Failure to comply with these cautions will void your warranty.

Step	Action
1	Select your cleaning solution (refer to page 23 for solution effects on metals).
2	Allowing for the volume of the parts you will be cleaning and cleaning solution, fill the tank with warm tap water to the operating level line (one inch from the top).
3	Add cleaning solution to the tank water.
4	Plug the cleaner into a grounded outlet.
5	For maximum efficiency, refer to page 16, "Optimizing Your Cleaner" before proceeding.

NOTE:

If this is the first time you are running the cleaner, or if you have changed cleaning solution, you must degas the solution. If not, skip to *"Cleaning Items"*.

Degassing		
Step	Action	
1	Turn the HEAT ON (MTH Cleaner only).	
2	Turn the TIMER to 5-10 and let the cleaner run to allow the solution to "degas". NOTE: Refer to page 17 for information on degassing.	

Cleaning Items

NOTE: To stop ultrasonics at any time, turn the TIMER to zero.

Step	Action
1	Set the TIMER for the amount of time you wish the items to be cleaned.
2	Place the items into a basket, perforated tray, or beakers in a positioning cover.
3	If using beakers or a solid tray, add cleaning solution to beakers or tray to cover the items.
4	Slowly lower the tray or beakers into the tank. Do not allow items to contact the tank bottom. Do not stir the solution.
5	When items are clean, slowly remove them from the cleaner.
6	Rinse the clean items with clean water and dry them, if necessary.

Operating Your DTH Cleaner



Explanation of Controls

Control	Function
POWER	Press to activate/deactivate power to the cleaner.
ON/OFF	After you press SELECT OPTION and set the LED Display for the selected option, press to activate HEAT (SET TEMP), SONICS (SET SONICS) and/or DEGAS (SET DEGAS).
LED Display	Indicates the tank temperature, set temperature, ultrasonics time or degas time setting, depending on your SELECT OPTION choice.
SET/ CLEAR DISPLAY	Used in conjunction with SELECT OPTION to set or clear the LED display. Press CLEAR DISPLAY to clear the LED display to 00. Press SET DISPLAY to reach your selection
SELECT OPTION	When pressed, toggles through the Function Indicators. This allows you to check or set the tank temperature and set ultrasonic cleaning or degas time.

Continued...

Control	Function
Function	Lights indicate the option selected by pressing SELECT
Indicators	OPTION.
	SOLUTION TEMP: Displays current solution temp. (10 -
Survey and the	75°C, ± 4°C).
and the states	SET TEMP: Set tank temperature (01 - 69°C).
	SET SONICS: Set ultrasonic time (01 - 99 mins., 60 mins. default).
and the second	SET DEGAS: Set degas time (01 - 99 mins., 5 mins. default).
	HEAT ONE: Indicates heat is activated and has been set (SET TEMP).

Before You Start Cleaning

Step	Action	
1	Select your cleaning solution.	
2	Allowing for the volume of the parts you will be cleaning and for the cleaning solution, fill the tank to the operating level line (one inch from the top) with warm tap water.	
3	Add a cleaning agent to the tank water.	
4	Plug the cleaner into a grounded outlet.	
5	Turn the POWER switch On. The cleaner will run through a three-second self-test. Wait until the LED Display shows 05 and the SET DEGAS Function Indicator lights.	

NOTE:

If this is the first time you are running the cleaner, or if you have changed cleaning solution, you must degas the solution. If not, move to *Setting Operating Parameters*.

Degassing

Step	Action
1	Degas for 5-10 minutes. If necessary, use SET/CLEAR DISPLAY to alter this setting. Default degas time is 5 minutes. NOTE: Refer to page 17 for information on degassing.
2	Press ON/OFF once to start the degas process.
3	After completing the degas time, you are ready to set operating parameters.

Setting Operating Parameters

Step	Parameter	Action
1	Set Time	The cleaner is now in Set Time mode with a default time of 60 mins. If necessary, use SET/CLEAR DISPLAY to alter this setting. Press ON/OFF once to activate timed ultrasonics.
2	Set Temp.	To set the tank temperature, press SELECT OPTION until the SET TEMP LED lights. Then press SET DISPLAY to alter the setting until the LED display indicates the tank temperature you wish to maintain. Press ON/OFF once to activate heat. The heat indicator lights.
3	Solution Temp.	To monitor the solution temperature, press SELECT OPTION until the SOLUTION TEMP LED lights. The LED display will indicate the actual temperature of the solution.

Cleaning Items

- Do not place parts or containers directly on the bottom of the cleaning tank; use a tray or wire to suspend items.
- Do not allow the solution to drop more than 3/8 inch below the operating level line with the cleaner on.
- Do not ever use alcohol, gasoline or flammable solutions. Doing so could cause a fire or explosion. Use only water-based solutions.
- Do not ever use mineral acids. These could damage the tank.

Failure to comply with these cautions will void your warranty.

NOTE:	Select Set Time then press ON/OFF once to stop ultrasonics at
	any time.

Step	Action
1	Place the items into a basket, perforated tray, or beakers in a positioning cover.
2	If using beakers or a solid tray, add cleaning solution to beakers or tray to cover the items.
3	Slowly lower the tray or beakers into the tank. Do not stir.
4	Press ON/OFF once to activate ultrasonics.
5	When the items are clean, press ON/OFF once to deactivate ultrasonics, then slowly remove the items from the cleaner.
6	Rinse clean items with clean, warm water and dry, if necessary.

- **To repeat a timed cleaning cycle** press ON/OFF once while in the Set Sonics mode. This cleaning cycle time will remain in memory until reset or you turn off the power to the cleaner.
- To reset ultrasonics time during a cleaning cycle press ON/OFF once, press SELECT OPTION until the SET SONICS LED lights. To increase time, press SET DISPLAY to your desired setting. To decrease time, press CLEAR DISPLAY, press SET DISPLAY to set the time, then press ON/OFF once to resume the cycle.
- To monitor the solution temperature press SELECT OPTION until the SOLUTION TEMP LED lights. The LED Display will display the solution temperature in degrees Centigrade (± 4°C). The cycle will continue during this process.

Draining Your Cleaner



 $\frac{1}{2}$ and $\frac{3}{4}$ Models do not have a drain. To empty, use the indented side of the rim to pour the used solution into a waste disposal unit, rinse the tank thoroughly and refill with new solution.



1-1/2, 2-1/2 and 5-1/2 gallon models include a drain and valve kit.



Optimizing Your Cleaner

Tanks

Cleaning - check the tank for contamination whenever you change solution. If necessary, remove contaminants with a nonabrasive cloth and water.

Emptying - always unplug the cleaner before emptying the tank. Empty the solution into a waste disposal unit.

Filling - always unplug the line cord before filling the tank. Fill the cleaner to the operating level (one inch from the top with beaker/tray in place), using warm tap water.

Low solution level - will cause the cleaner to fail. When you remove heavy or bulky loads from the cleaner, the solution level may drop below the operating level. If so, be sure to replace lost solution and degas, if necessary, depending on the amount used.

Overload - do not rest any items on the tank bottom. Weight on the tank bottom dampens sound energy and will cause damage to the transducer. Instead, use a tray and/or beaker positioning cover to support all items. Allow at least one inch between the tank bottom and the beaker or receptacle for adequate cavitation.

Covers - allow the cleaner to heat up faster, to a higher temperature, and avoid excessive liquid evaporation. However, obstructing the cover vents will cause the cleaner to overheat.

Temperature

Heater - the heater may cause some discoloration of the tank wall. This is normal and will not affect the performance of the unit.

Solution - the fastest method to heat your cleaner is to fill with warm solution, use heat, ultrasonics (which also adds heat), and a cover.

Approximate stabilization temperatures with ultrasonics and heat running continuously:

MTH/DTH cleaner without a cover 50° MTH/DTH cleaner with a cover 62°

continued ...

Over temperature protection (DTH only) - the cleaner will shut down at 75°C and the LED display will blink "75". Turn the cleaner off and allow it to cool down. For a faster cooldown, replace some of the warm solution with cold solution.

Solution

Solution activity - the amount of visible activity is not necessarily related to optimum cavitation for cleaning.

Degassing - fresh solutions contain many dissolved gases (usually air), which reduce effective ultrasonic action. Although solutions will naturally degas over time, using Degas mode speeds up the degassing process. Solutions that have been sitting unused for 24 hours or longer have reabsorbed some gases.

Heat - increases the chemical activity of cleaning solutions.

Surface tension - can be reduced by adding solution to the bath. Reduced surface tension will increase cavitation intensity and enhance cleaning.

Solvents - never use solvents. Vapors of flammable solutions will collect under the cleaner, where ignition is possible from electrical components.

Renewal - replace cleaning solutions often to increase ultrasonic cleaning activity. Solutions, as with most chemicals, will become depleted over time. Solutions can become contaminated with suspended soil particles which coat the tank bottom, inhibiting ultrasonic activity.

Application Hints

- Never clean *novelty or inexpensive jewelry* in the cleaner. The combination of heat and vibration may loosen a cement-held setting.
- Never clean *gemstones* such as emerald, amethyst, pearl, opal, coral, turquoise, peridot or lapis lazuli in the cleaner.

First time cleaning - first experiment with one piece, then proceed with the remainder.

Solution level - Be sure to maintain solution level within 1/2 inch of the tank's "operating level" line. Surface activity can vary with liquid level.

Load size - It is faster and more efficient to run several small loads rather than a few big loads.

Placing items - Never allow items to sit on the bottom of the tank. Always place them in a tray or beaker or suspend in the solution.

Rinsing items - After cleaning, use a clean water bath to rinse away chemicals adhering to items.

Lubricating items - When necessary, re-lubricate items immediately after cleaning.

Drying items - Air drying at room temperature works for some items. Place parts requiring faster drying time under hot air blowers or in ovens.

Please call your local distributor if you have application questions.

Cleaning Methods

There are two methods of cleaning - direct and indirect. Each has advantages and disadvantages. When in doubt, run test samples using both methods to decide which one produces the best results for you.



How it works:

- Fill the tank with warm water and a cleaning solution.
- Place the items to be cleaned in a perforated tray and lower them into the tank. You can also suspend items on a wire and then immerse them in the solution.

The advantages of this method are the simplicity of operation and cleaning effectiveness.

Indirect Method



How it works:

- Fill the tank with warm water and a cleaning solution.
- Pour your solution medium into one or more beakers or into a solid insert tray.
- Place the beakers in a beaker positioning cover or a solid insert tray to fit your cleaner. Beakers should not touch the tank's bottom.

The advantages of this method are:

- Removed soil stays in the beaker or tray so you can easily examine, filter or discard it.
- You can use one or more solutions at the same time.
 - two completely different cleaning solutions.
 - one beaker or tray with a cleaning solution and one with a rinse solution.
- Cleaning solution in your tank needs to be changed less often.

Cleaning Solutions

Do not use alcohol, gasoline, bleach, mineral acids, solutions with a flash point, semi-aqueous or combustible liquids in ultrasonic tanks, or you will void the warranty. Only use non-flammable solutions and water-based solutions.

Solution Types

Water-based solutions can be either slightly acidic or alkaline. They include detergents, soaps and industrial cleaners designed to remove specific soils.

Acidic water-based solutions: remove rust, tarnish or scale. They range from mild solutions that remove tarnish, to concentrated, inhibited acidic solutions that remove investment plaster, milk-stone, zinc oxide and rust from steel and cast iron as well as smut and heat-treat scale from hardened steel.

Alkaline water-based solutions: include carbonates, silicates and caustics. These cause emulsifying action, which keeps soil from redepositing on the cleaned surface, and improves cleaning action in hard water.

Alkaline strength	Removes:
Mild	Light oils and greases, cutting oils and coolant compounds.
Mild to strong	Heavy grease and oil, waxes, vegetable oils, inks, wax or fat-base buffing and polishing compounds, milk residues and carbohydrates.
Heavy-duty	Mill scale, heat-treat scale, corrosion or oxides.

Change the cleaning solution periodically. Cleaning solutions can become contaminated with suspended soil particles which coat the tank bottom. This coating dampens the ultrasonic action and reduces cleaning efficiency. Certain solutions will cavitate better than others. Contact your local distributor for further information.

Heat and cavitation increase the chemical activity of cleaning solutions. Some materials may be damaged by this stronger chemical action. When in doubt, test run samples of items to be cleaned.

Caustic solutions: used to remove rust from steels, metal alloy corrosion and a variety of tenacious soils.

Solution Amounts

Solution amounts may vary. The amount you use depends on the detergent and the type of soil to be removed. Follow instructions on the solution container and refer to the table below for the effects of solutions on metals.

Chemicals Harmful to Your Tank

The following chemicals will harm your ultrasonic tank and the action of ultrasonics and higher operating temperatures will increase their chemical activity. Do not use these or similar chemicals directly or in dilution in your ultrasonic tank or you will void your warranty.

Acetophenone Aluminum Chloride Aluminum Fluoride Aluminum Sulphate Ammonium Bifluoride Ammonium Chloride Ammonium Hydroxide Amyl Chloride Antimony Trichloride Aqua Regia Bromine Calcium Bisulfate Calcium Bisulfite Calcium Hypochloride Chloracetic Acid Chloric Acid Chlorine, Anhydrous Chromic Acid Copper Chloride Copper Fluoborate Ethyl Chloride Ferric Chloride Ferris Sulfate Fluoboric Acid Fluorine Hydrobromic Acid Hydrochloric Acid

Hydrocyanic Acid Hydrofluoric Acid Hydrofluosilicic Acid Iodoform Mercuric Chloride Muriatic Acid Phosphoric (crude) Sodium Hypochlorite Potassium Chloride Stannic Chloride Stannous Chloride Sulfur chloride Sulfur chloride Sulfuric Acid Zinc Chloride

Solution Effects on Metals

Cleaning Agent	Steel	Brass	Alumi- num	Magne- sium	Zinc	S. Steel Copper	Tin
Optical (1)	none	none	none	none**	none**	none	none**
Jewelry (1)	none	none	none	none	none	none	none
Buffing (1) compound	none	slight stain	none	none	attacks	none	none
Oxide (2) remover	slight etch	none	slight attack	attacks	attacks	none	none
Electronic cleaner (1)	none	none	slight attack	none	none	none	none
General(1) purpose	none	none	slight attack	none	none	none	none
Industrial strength(1)	none	none	slight attack	none	none	none	none
Metal (1) cleaner 1	none	none	none	none	none	none	none
Metal (1) cleaner 2	none	none	slight attack	none	none	none	none
Metal (1) cleaner 3	none	none	none	none	none	none	none
Liquid Rust (3) stripper	none	none	attacks	attacks	attacks	none	slight attack
GP (1) Powder	none	none	none	none	none	none	none

(1) = Alkaline; (2) = Acidic; and (3) = Caustic.

2 warning 2

*Free hydrogen may be released if solution comes in contact with reactive metals.

** No effect if solution temperature is less than 140°F.

Troubleshooting

If your cleaner does not operate satisfactorily, please check the tables below for possible causes before calling your authorized service center.

\triangle warning \triangle

High voltage inside - dangerous shock hazard. DO NOT attempt to disassemble or repair the cleaner.

Problem	Cause	What to do
Cleaner will not	Cleaner not plugged in properly.	Plug into functioning electrical outlet.
start.	<i>MT</i> - Mechanical timer not ON.	Turn timer clockwise.
	<i>DTH</i> - POWER switch not ON.	Press power switch ON.
	DTH - Start button malfunctioning.	Call nearest authorized service center.
	Blown fuse.	Call nearest authorized service center.
Cleaner operates	Heater malfunctions.	Call nearest authorized service center.
but does	MTH - HEAT not ON.	Turn heat ON
solution	<i>DTH</i> - HEAT not set properly.	See Operating Your DTH Cleaner.
	DTH - membrane malfunctioning.	Call nearest authorized service center.
Clogged drain	Clogged drain.	Call nearest authorized service center.

High voltage inside - dangerous shock hazard. DO NOT attempt to disassemble or repair the cleaner.

Problem	Cause	What to do
Cleaner	Malfunctioning heater or	Call nearest authorized
operates but	sensor components.	service center.
does not		
maintain set		
temperature		
Cleaner	Interrupted calibration	Press SET DISPLAY
operates but	sequence.	
display does	DTH - timer board	Call nearest authorized
not function.	malfunctioning.	service center.
Cleaner	Overheat condition.	Turn cleaner off. Allow
stops		cleaner to cool, check
operating		solution level, then restart.
and display		Refer to Optimizing Your
blinks "75".		Cleaner.
Decreased	Solution is not degassed.	Make sure that tank was
ultrasonic		filled with warm tap water
activity.		plus cleaning solution and
NOTE:		has run 5-10 minutes.
Refer to	Solution is spent.	Change solution.
cavitation	Solution level is incorrect	Adjust solution to within 3/8
check	for load.	inch of the tank's operating
onoon.		level line with load.
	Tank bottom is covered	Empty, then clean tank with
	with soil particles.	warm water Wine with a
		nonabrasive cloth.
	Using deionized water in	Deionized water does not
	the tank.	cavitate as actively as
		soapy tap water.

Check your cleaner periodically to test the level of activity of the ultrasonic cavitation. Frequency of testing will depend on your use of the cleaner, however, Branson suggests running this test monthly.

Glass Slide Test

You will need the following equipment:

- Frosted microscope glass slide (1" x 3"), such as ESCO #2951F, or equivalent;
- No. 2 lead pencil; and
- General purpose household cleaning solution, such as "Dawn" liquid soap.

Test procedure:

- 1. Prepare a fresh solution with general purpose household cleaning solution (concentration 1%) and warm tap water (120° 140°F).
- 2. Fill the cleaner to within 3/8 inch of the "operating level" line.
- Turn the ultrasonics on for at least five to ten minutes to allow for degassing.
- 4. Prepare the glass slide by first wetting the frosted portion with tap water.



- 5. With the No. 2 pencil, on the frosted portion make an "X" from corner to corner.
- 6. Immerse the frosted end of the slide into the solution. Hold the slide vertically and center it in the solution.
- 7. Make sure that model DTH cleaners are in SET SONICS mode, not degas mode, then turn ultrasonics On.

The ultrasonics will begin immediately to remove the lead from the slide. All lead should be removed within 10 seconds. If your cleaner passes this test, its ultrasonic cavitation is acceptable.

NOTE:

To ensure consistency from test to test, be sure to repeat test conditions - use the same solution concentration, liquid level, temperature, type of pencil, length of degassing, etc.

KIMBLE KONTS ZS ml HF

Service Center

With normal use, your Ultrasonic Cleaner should not require servicing. However, if it fails to operate satisfactorily, first try to diagnose the problem by following the suggestions in the Troubleshooting Guide.

You will void the warranty if you disassemble your cleaner. High voltage inside the cleaner is dangerous.

If you find that your cleaner needs repair, carefully pack and return it to your local distributor. If under warranty, remember to include proof of purchase.

Your cleaner will be shipped by ground service unless you specify otherwise.

Authorized Service Centers

Name	Address	Tel/Fax Number
Alpha Omega Electronics Corp.	2821 National Drive Garland, TX 75041	Tel: 972-271-5571 Tel: 800-540-4967 Fax: 972-840-3668
Crystal Electronics Inc.	1251 Gorham St. Unit 2 Newmarket, ON Canada L3Y 8Y6	Tel: 905-953-9129 Fax: 905-953-7965
Paragon Electronics	6861 SW 196 th Ave. Suite 404 Pembroke Pines, Florida 33332	Tel: 954-434-8191 Fax: 954-434-8385
Master Sonics Repair Center	77 Whiting St. Plainville, CT 06062	Tel: 860-410-1700 800-737-2198 Fax: 860-410-1704

Technical Support

Branson Ultrasonics Corp.	41 Eagle Road P.O. Box 1961 Danbury, CT 06813-1961	Tel: 203-796-0339 Tel: 203-796-0557 Tel: 800-732-9262
	1	Fax: 203-796-2240

PROCESS EQUIPMENT & SUPPLY, INC. 31255 Lorain Road Cleveland, Ohio 44070 1-800-539-6336 • Fax 1-800-539-6371 WWW.PROEQUIP.COM





Read this manual thoroughly before operating the VWR Labmax Bottle Top Dispenser !

General Safety Precautions

When using the VWR Labmax please observe the following safety precautions

- Follow all safety regulations. Always wear eye protection, gloves and other necessary protective clothing
- Observe any safety instructions and precautions on the reagent bottles
- All users should read and follow this manual carefully
- Keep this manual easily available to all users
- The Labmax is designed for dispensing liquids only in accordance with its operating limitations
- If unsure about dispensing a specific solution, please contact VWR International at 1-800-932-5000.
- Always dispense liquids away from the operator and other persons
- Avoid splashes!
- Volatile substances must be dispensed in a fume hood
- Never carry the dispenser/bottle assembly by the dispenser
- Never carry the mounted instrument by the cylinder sleeve
- Regularly inspect the VWR Labmax and dispenser tube for leakage
- Clean the VWR Labmax regularly to maintain proper operation
- Ensure the suction and ejection cannulas are firmly in position before using the VWR Labmax
- Use extra caution when dispensing corrosive, poisonous, radioactive or hazardous chemicals

Upon receipt of the VWR Labmax Dispenser inspect the instrument for any damage that may have occured during transit. Any damage must be reported to the carrier within 48 hours.

Optional Accessories

The following optional accessories are available for the VWR Labmax Dispenser.

- Borosilicate Glass Check Valve.

This special, plastic-safety coated check valve is to be inserted into the valve axle (10) on the side opposite to the cannula and prevents volatile reagents from escaping through the ventilation canal. The Glass Check Valve is recommended when using your dispenser for bromine.

- Borosilicate Glass Calcium Chloride Tube.

This safety coated tube can be filled with calcium chloride and will protect drying agents from external humidity.

Disposable Filters are available to protect against dust and bacteria. The filters fit into the valve axle socket.

Threaded Bottle Adapters

Catalog #	Description	Thread Size
40000-084	Threaded Bottle Adapter (pack of 3)	25mm
40000-082	Threaded Bottle Adapter (pack of 3)	28mm
40000-074	Threaded Bottle Adapter (pack of 3)	32mm
40000-080	Threaded Bottle Adapter (pack of 3)	38mm
40000-078	Threaded Bottle Adapter (pack of 3)	40mm
40000-076	Threaded Bottle Adapter (pack of 3)	45mm
40000-072	Combination Pack	
	(1each: 25mm, 28mm, 32mm and 45r	nm)

To reorder contact VWR International 1(800) 932-5000



Common Spare Parts

Suction Tube (14)	2 - 10 ml	D-5375-000
Suction Tube (14)	10- 50 ml	D-5375-001
Ejection Cannula	2 - 10 ml	D-5375-003
Ejection Cannula	10 - 50 ml	D-5375-004
Extended Discharge	Tube, 80cm for 2.5ml to 10ml	D-5375-002
Extended Discharge	Tube, 80cm for 25ml to 100ml	D5375-012

Warranty

VWR Scientific Products warrantees the VWR Labmax you have received to be free of defects in material and workmanship for 24 months from the date of purchase. VWR's responsibility shall be limited to the repair or replacement of the VWR Labmax at VWR's option.

This warranty is valid only if the VWR Labmax is used in the manner described in this manual and for the purpose for which it is designed.

VWR International shall not be responsible for consequential damages resulting from the misuse of this equipment.

Operating Limitations

The VWR Labmax is suitable for dispensing most liquids. Extra care should be taken when dispensing dangerous or hazardous solutions.

The VWR Labmax Dispenser should not be used for dispensing the following

- solutions of hydrofluoric acids
- solutions which contain or form solids
- solutions of substances catalyzed by platinum and iridium alloys

The VWR Labmax should be rinsed daily if used with the following :

- solutions which form crystals
- inorganic oxidizing solutions (i.e. biuret reagent)

The temperature of the VWR Labmax Dispenser and reagent should not exceed $40^{\circ}C$ ($104^{\circ}F$).

Assembly

Select the appropriate length of suction tubing for the size bottle being used.

Press the tubing firmly into position on the underside of the VWR Labmax cap. Screw the ejection cannula on firmly.

Attention :

Do not move the plunger before the dispenser has been completely assembled.

The VWR Labmax Dispenser will fit on a variety of bottle tops using the various supplied adapters.

The VWR Labmax Dispenser should be firmly screwed onto the threads of the bottle from which liquid is to be dispensed.

When placing the VWRbr Labmax on the bottle top hold it by the base and not by the glass cylinder.

Safe and proper operation is only possible with the ejection cannula in the condition it is originally supplied.

Never attempt to operate the VWR Labmax with a damaged or deformed cannula. Do not use any other type of tubing. Regularly inspect the tubing for leakage and immediately replace any damaged or deformed tubes.

Hold the housing at the base when screwing or unscrewing the instrument rather than gripping. Never carry the dispenser on its housing! The various adapters that come with the VWR LABMAX will allow the dispenser to fit on many different bottles.

Dispensing

Be sure to follow all safety instructions. Always wear eye protection and other appropriate safety equipment. Place the vessel that solution is to be dispensed into under the ejection cannula. The ejection cannula must point away from the user and other persons at all the times.



Ejection Cannula

Disassembly

WARNING:

The dispenser must be removed from the bottle and must be completely empty before attempting to disassemble.

- 1. Adjust the Dispenser to the maximum volume setting.
- 2. Raise the outer housing (6) and pull off the adapter ring.
- 3. In order to remove the quick-volume adjustment switch (4) out of the outer housing (6), move it downward.
- 4. If it is necessary to remove the PTFE plunger (3), this can be done by unscrewing the blue fix screw (2) on the top of the Dispenser.
- 5. Loosen the cap nut (8) and remove the upper portion of the unit (glass tube/PTFE-cap) paying special attention to the slot on the PTFE cap (7) and also using caution not to lose the small spring and ceramic bead which are set into the valve head. During reassembly, this cap must be positioned correctly in relation to the valve head (9).
- 6. To disassemble the valve axle (10) unscrew the over-twist device (12), so that the axle can be pulled out of the valve head. Remove the axle by pulling while twisting to the left and right.
- 7. To remove the calibrated glass cylinder from the PTFE-cap, gently pull them apart or heat the parts in a boiling water bath if they are stuck. During assembly observe that the glass cylinder is pressed firmly into the bottom of the cap. If the parts have been heated in a water bath, let them cool down before reassembling.
- 8. The removal of damaged valves should only be done by an authorized service technician.

Please refer to diagram on next page.

WARNING:

Always wear protective clothing, gloves and safety goggles when disassembling the VWR Labmax dispenser.

Prevention of sticking valves during prolonged intervals

When the VWR Labmax Dispenser is screwed onto a bottle be sure that the valves are surrounded by liquid.

When the VWR Labmax is not on a bottle, smooth running of the valves can be maintained by rinsing with distilled water and/or laboratory detergent. After rinsing draw ethanol through the Dispenser.

To ensure the accuracy of the VWR Labmax, test with 2 x distilled water on a semi-micro scale.

Remember to allow for temperature dependence (1 ml water at $20^{\circ}C = 0.998g$).

Sterilization

After removal of the suction tube and ejection cannula the VWR Labmax can be autoclaved at 121° C and maximum of 2 bar. Place the VWR Labmax in an upright position on a towel. Contacts with hot metal surfaces should be avoided.

IMPORTANT:

Before autoclaving, adjust the volume of the VWR Labmax to 2/10 of its total volume.

Caution:

Let the VWR Labmax cool slowly after autoclaving.Do not use the VWR Labmax until it has cooled to room temperature. Inspect all parts and tubing for leakage after sterilization.

It is also possible to sterilize the VWR Labmax chemically with ethanol or other sterilizing reagents.

Warning:

Avoid personal injury from chemicals. To protect yourself wear eye protection and other appropriate safety equipment and clothing. Please follow all safety instructions and observe operating procedures.

Safe and proper operation is only possible with the ejection cannula as originally supplied. Never use damaged or deformed cannulas. Do not use any other type of tubing. Regularly inspect the tubes for leakage and immediately replace damaged or deformed ones.

Warning:

Never press on the plunger without a collection vessel located under the ejection cannula !

CAUTION: If the valve is damaged, reagent may drip from the ejection cannula !

To remove air

The drip-free ejection cannula shows the position of the valve axle. To purge the air from the system, turn the valve axle clockwise 90°, against the stop.Press the plunger to remove air (This may take several pump strokes !).

During this process no reagent will be lost as the axle is in position to make a closed circuit.



Ejection Cannula

Volume adjustment

The guick volume adjustment has been designed for reproducibility in dispensing.

The volume is adjusted by pressing the upper part of the volume rocker switch.

The lower part of the switch will then be disengaged from the toothed system and the volume easily adjusted.

Slide the switch up or down until the indicator on the left side is pointing to the appropriate volume on the scale.

Release the rocker switch. Turn the ejection cannula away from you and place a vessel underneath it.

The valve axle must now be turned to the dispensing position (cannula spout facing downward).

Raise the plunger slightly and press down to fill the ejection cannula with solution.

Fill it very carefully to the tip.

Raise the plunger slowly until it stops.

The Dispenser is now filled.

Gently depress the plunger to dispense the liquid into the collection vessel.

Attention :

Never force the plunger downwards. Raise and depress the plunger slowly. Always leave the plunger down and empty after use.

Rest Position

Turn the axle valve to the left (180°) until it stops. This will close the dispensing position. Tip the dispenser/ bottle assembly to allow the solution in the ejection cannula to flow back into the bottle. Any reagent left in the Dispenser will flow back into the bottle. The cannula will not drip and the need for a cap on the ejection cannula is eliminated.

No reagent will be lost while the VWRbrand Labmax is not in use and no reagent will leak into the environment.

Ejection Cannula



Cleaning and maintenance

The VWR Labmax Dispenser can be disassembled quickly and easily. Cleaning the dispenser is as easy as the replacement of parts, if required.

To ensure proper performance and a long life the VWR Labmax should be cleaned according to the following schedule :

- When changing reagents
- Before storing for a long period of time _
- Daily if dispensed any of the following :
 - substances which form crystals
 - alkaline solutions
 - organic solvents, inorganic oxidizing solutions (i.e. biuret reagent)

Preparation for cleaning

- 1. Reagent remaining in the VWR Labmax should be returned to the reagent bottle by turning the valve axle 90° clockwise and pumping the plunger.
- 2. Place the dispenser/bottle assembly in the sink.
- 3. Unscrew the VWR Labmax from the bottle and lift so that the suction tube is no longer immersed in liquid.
- 4. Tap the suction tube carefully on the inside of the bottle to remove any solution remaining in the tube.
- 5. Lift the Dispenser from the bottle and rinse with distilled water.

The VWR Labmax can now be disassembled and cleaned according to the instructions in the disassembly section of this manual.

WARNING:

The VWR Labmax suction and ejection tube may contain liquid. Always point them away from your body. Wear eye protection and protective clothing.

Brinkmann Bottletop Dispenser and ChemSaver™ Bottletop Dispenser

Instruction Manual Mode d'emploi Manual de Instrucciones Istruzioni d'impiego Bedienungsanleitung

Components

Brinkmann Bottletop Dispenser

Distributeur standard pour flacons Brinkmann Dispensador de frasco Brinkmann

Brinkmann Dispensatore per flaconi

Brinkmann Flaschendispenser



- 1 Fine volume adjustment knob
- 2 Piston holder
- 3 Cylinder sleeve
- 4 Volume setting knob
- 5 Piston
- 6 Glass cylinder
- 7 Protective cylinder sleeve
- 8 Discharge valve toggle
- 9 Discharge tube sleeve
- 10 Discharge tube
- 11 Discharge valve for Brinkmann ChemSaver™ Bottletop Dispenser

Brinkmann ChemSaver™ Bottletop Dispenser Distributeur pour flacons Chemsaver ™ Brinkmann Dispensador de frasco Brinkmann ChemSaver™ Dispensatore per flaconi Brinkmann ChemSaver™ Flaschendispenser



- 12 Discharge valve for Brinkmann Bottletop Dispenser
- 13 Cap for discharge tube
- 14 Valve block housing
- 15 Valve block (inside housing)
- 16 Valve ball
- 17 Filling valve
- 18 Air vent cover / filter connection
- 19 Cylinder ring (inside cylinder sleeve)
- 20 Tool:
 - a) For sizes 1/2.5/5/10 mL
 - b) For sizes 25 / 50 mL
 - c) Enlarging ring for size 100 mL

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1 Safety Precautions

- This manual does not purport to address all the safety problems associated with the use of this dispenser. It is the responsibility of the user to consult and establish appropriate safety and health practices and determine applicability of intended use.
- Observe extreme caution when dispensing caustic, poisonous, radioactive or hazardous chemicals.
- Observe general safety regulations for laboratory hazard prevention (eg. wear protective clothing, gloves, and glasses).
- Follow the reagent manufacturer's safety information.
- Every user must be acquainted with this instruction manual and have it readily available at all times.
- Use the instrument only for its proper purpose and within the limits stated in the Operating Limitations. If in doubt as to the suitability of your application or reagent, please be sure to consult Brinkmann Instruments.
- Regularly inspect the instrument for leakage and signs of wear. Before use, make sure all fittings and the connection to bottle are secure.
- Never use force on the instrument. Pull the piston up and press it down gently.
- Make sure the dispensing tube is facing away from the user or other persons when operating. Avoid splashes. Dispense only into suitable vessels.
- Do not dispense without discharge tube sleeve.
- Do not carry a mounted instrument by the cylinder. Always support both the instrument and the reagent bottle.
- Do not depress the piston when the cap for the discharge tube is in place.
- Use only original manufacturer's parts and accessories.
- If an instrument is not operating properly, immediately stop dispensing. Clean and repair the instrument according to the instructions in this manual or contact Brinkmann Instruments.

2 Contents of Package

The following items are included with every Dispenser

- 1 Dispenser
- 2 28 mm adapter
- 3 38 mm, 45 mm adapter with 1 mL, 2.5 mL, 5 mL, 10 mL dispensers, or 33 mm, 38 mm adapter with 25 mL, 50 mL, 100 mL dispensers
- 4 Manual
- 5 Telescoping filling tube
- 6 Assembly / Disassembly Tool
- 7 Performance Documentation on back page of manual

Verify that you have received all the items listed. If you are missing pieces, contact Brinkmann Instruments. Notify the supplier you purchased the instrument from at once if you find the instrument has been damaged in shipment.

3 Components

(Please open up the fold-out cover at the front of this manual)

4 Design Principle

The Brinkmann Bottletop Dispensers are designed for dispensing liquids in the milliliter range directly from a reagent bottle.

When the instrument is used correctly the dispensed reagent comes into contact with the following chemically resistant materials: PFA, Borosilicate glass, Platinum Iridium, ETFE, PTFE, FEP.

Volume range	Setting increments	Dead volume	<i>Systematic</i> <i>error</i> (Inaccuracy)	<i>Random</i> <i>error</i> (Imprecision; CV)
0.50 – 2.5 mL	0.05 mL	0.21 mL	± 0.6 %	≤ 0.1 %
1.00 – 5.0 mL	0.10 mL	0.18 mL	\pm 0.5 %	≤ 0.1 %
2.00 - 10.0 mL	0.20 mL	0.17 mL	± 0.5 %	≤ 0.1 %
5.00 – 25.0 mL	0.50 mL	1.20 mL	± 0.5 %	≤ 0.1 %
10.00 – 50.0 mL	1.00 mL	1.31 mL	± 0.5 %	≤ 0.1 %
20.00 – 100.0 mL	2.00 mL	1.21 mL	± 0.5 %	≤ 0.1 %
1 mL Fixed	-	0.21 mL	± 0.4 %	≤ 0.3 %
5 mL Fixed	-	0.18 mL	± 0.5 %	≤ 0.1 %
10 mL Fixed	-	0.17 mL	± 0.5 %	≤ 0.1 %

5 Specifications (Includes all dispensers)

Liquid:Bidistilled waterReference Temperature:20 °C, constantNumber of determinations:10,
according to ISO 8655-6

Note: Specifications are given for maximum volume.

Technical specifications subject to change!

Materials

	Brinkmann Bottletop Dispenser	Brinkmann ChemSaver™ Bottletop Dispenser
Piston Holder	PP	PP
Cylinder Casing	PP	PP
Volume Adjustment Knob	PPN GV2/30	PPN GV2/30
Valve Block Housing	PP	PP
Valve Block	PTFE	PTFE
Air Vent Cap	PP	PP
Telescopic Filling Tube	FEP	FEP
Discharge valve	ETFE	PFA
Discharge Tube	PFA	PFA
Discharge Tube Support	PP	PP
Discharge Tube Cap	PVDF	-
Spring for Discharge Valve	Pt- Ir	Pt- Ir
Valve Balls	Borosilicate (Duran) Glass	Borosilicate (Duran) Glass
Filling Valve	ETFE	ETFE
Cylinder	Borosilicate (Duran) Glass	Borosilicate (Duran) Glass
Protective cylinder sleeve	PTFE	PTFE
Piston 1 – 10 mL 25 – 100 mL	ETFE Duran Glass	ETFE Duran Glass
Piston Seal	PTFE	PTFE
6 Operating Limitations

The instrument is suitable for most media with the following exceptions:

- Hydrofluoric acid solutions
- Solutions which tend to crystallize, contain or form solid particles
- Unstable substances which react catalytically with Platinum-Iridium (e.g. $H_2 O_2$)
- Liquids attacking bosilicate glass, PFA, ETFE, PTFE, Platinum- Iridium, FEP
- Oxidizable inorganic solutions which may precipitate metal oxides (e.g. Biuret reagent)

Recommended temperature range for operating the instrument and for reagent is 15 °C to 40 °C. Do not exceed these temperatures.

The user is responsible for verifying the suitability of the Brinkmann Bottletop Dispenser for his/her application. This includes verifying chemical compatibility of the reagent to be used.

7 Assembling the Instrument

7.1 Attaching threaded adapters

- The 1 mL, 2.5 mL, 5 mL, and 10 mL dispensers will directly attach to reagent bottles with a 33 mm threaded neck.
- The 25 mL, 50 mL and 100 mL dispensers will directly attach to reagent bottles with a 45 mm threaded neck.
- For bottles with different size necks, measure the inner diameter of cap for the reagent bottle. Choose the size adapter which best matches this diameter and thread it onto the reagent bottle.

Note: Be sure the adapter fits securely onto the bottle threads.



7.2 Connecting the filling tube (Fig. 1)

For dispenser 1, 2.5, 5.0 and 10.0 mL:

 Push the smaller diameter end of the filling tube onto the filling valve (17) as far as possible.

For dispensers 25, 50 and 100 mL:

 Push the larger diameter end of the tubing onto the filling valve.

Note: Be careful not to bend the tube.





- Determine the necessary length of the telescopic filling tube by measuring the distance from the bottom of the bottle to the top of the threaded base (Fig. 2).
- Extend the telescopic filling tube to the desired length and cut the end of the sleeve on an angle.

Note: If the bottle is shorter than the the filling tube, separate the two sections and cut the approriate piece to the necessary length.



Caution: When hygroscopic fluids are being used, a dry tube can be connected to the Dispenser. To do so, remove deairing cover (11) and push the dry tube into the opening. The dry tube can be filled with a moisture absorber (e.g. silica gel, 1 - 3 mm particle size) or CaCO₂. To absorb CO₂, fill the tube with NaOH pills (5 mm dia.)

8 Connecting to the Bottle



 Holding the threaded base, mount the instrument onto the bottle or adapter by turning. Be sure the dispenser fits tightly onto the bottle.

Caution: Do not move the piston until the instrument is completely mounted.

Fig. 3



Warning: Do not carry the mounted instrument by the upper casing. Always support both the dispenser and the bottle. Carry the mounted instrument only as shown in the figure.



9 Dispensing



Fig. 5

9.1 Removing air from the instrument (Fig. 5)

Caution: The discharge tube must always point away from the user and others at all times.

For Brinkmann ChemSaver™ Bottletop Dispensers

Caution: Do not move the piston until the instrument is completely mounted and the discharge valve is in the recirculate position (\bigcirc).

- Turn the discharge valve into the recirculate position by turning the toggle until it points to the recirculate symbol () on the side of the discharge tube support sleeve.
- Place a collecting vessel below the discharge tube.

Caution: Be careful of chemicals dripping off the discharge tube.

 Raise the piston gently up approximately 2 cm and press down firmly. Repeat this movement until no more bubbles appear in the cylinder and discharge tube.

Turn the discharge valve into the dispensing position by turning the toggle so it lines up parallel with the discharge tube support sleeve and points to the arrow symbol (\rightarrow). Raise the piston up slightly and down slowly until liquid appears at the end of the discharge tube.

For Brinkmann Bottletop Dispensers

 Place a collecting vessel below the discharge tube.

Remove stopper cap from the discharge tube.

Caution: Be careful of chemicals dripping off the discharge tube and stopper cap.

 Raise the piston gently up approximately 2 cm and depress down firmly. Repeat this movement until no more bubbles appear in the cylinder and discharge tube.



Fig. 6

9.2 Setting the volume

- Hold the cylinder casing down (Fig. 6).
- Slide the volume setting knob to the right and move it up or down to adjust to the desired volume.
- When the volume is selected, slide the volume setting knob to the left to lock it into place.



Fig. 7

9.3 Dispensing (Fig. 7)

 Place an appropriate collecting vessel below the opening of the discharge tube.

For Brinkmann Bottletop Dispenser

Remove the cap from the discharge tube.

Caution: Be careful of chemicals dripping off the discharge tube and stopper cap.

- Raise the piston slowly and evenly to the stop to pull reagent into the glass cylinder.
 - Push down slowly and evenly to expel the reagent into the collecting vessel.

Avoid splashing the reagent!

Note: Never use force to move the piston!

 When not in use leave the piston in the down position and push the cap onto the discharge tube.

Caution: After completing the dispensing operation for the ChemSaver[™] Bottletop Dispenser, always move the discharge valve into the recirculate position to prevent inadvertent dispensing from the discharge tube.

10 Cleaning

In order to maintain safe, accurate and precise operation, the dispenser must be cleaned in any of the following situations:

- Immediately when the piston action becomes difficult to move.
- When changing the reagent.
- Prior to long term storage.
- Prior to any maintenance or repair.
- Daily after use with reagents listed under operating limitations (e.g. solutions prone to crystallization, alkaline solutions, organic solvents, oxidizable inorganic solutions).
- Prior to sterilization.

10.1 Preparations for cleaning

Caution: Wear gloves, saftey glasses and a lab coat. Observe procedures for safe handling of hazardous reagents.

- If you are operating a ChemSaver[™] Bottletop Dispenser, turn the valve to the recirculate position (<a>C).
- Be sure the piston is in the down position, and the cap is securely on the end of the discharge tube of the Brinkmann Bottletop Dispenser.
- Place the mounted instrument into a sink or appropriate container.
- Adjust the volume to the maximum setting.



Fig. 8





- Holding the threaded base, disconnect the instrument from the bottle by turning it counterclockwise at the threaded base. Lift the instrument up far enough so that the filling tube is no longer immersed.
- Carefully tap the filling tube against the bottle from the inside so that the reagent runs back into the bottle (Fig. 8).
- Lift the instrument from the bottle.
- If you are operating a ChemSaver™ Bottletop Dispenser, move the discharge valve toggle into the dispensing position (→).
- Remove the cap from the discharge tube of the Brinkmann Bottletop Dispenser.
- Hold the instrument so that the discharge tube is over the bottle opening and dispense the remaining reagent back into the bottle (Fig. 9).

Caution: During cleaning or changing of bottles, the discharge tube contains liquid even when the discharge valve on the ChemSaver™ Bottletop Dispenser is in the recirculate position.

10.2 Standard cleaning procedure

- Immerse the filling tube into a suitable neutral cleaning solution. Rinse by repeated dispensing of the maximum volume.
- Remove the dispenser from the cleaning solution and evacuate all solution from the unit.
- Place the filling tube into distilled water and rinse by repeated dispensing of the maximum volume.
- Lift the filling tube out of the distilled water and empty the instrument by repeated dispensing.

10.3 Intensive cleaning procedure

If the instrument becomes heavily soiled or extensive crystallization has formed, it may be partially disassembled for intensive cleaning. Refer to section 14 for disassembly and assembly procedures.

11 Sterilization

The entire instrument can be steam- sterilized at 121 °C, 15 psi (2 bar), for 20 min. Do not disassemble. The user must verify the efficiency of the sterilization.

- Clean the dispenser thoroughly following the procedures described under cleaning (sec. 10). Standard cleaning should be sufficient unless the instrument is heavily soiled.
- If you are operating a ChemSaver[™] Bottletop Dispenser, leave the discharge valve toggle (8) in the dispensing position (→).
- Disengage the volume setting button (4) by moving it to the right. Push it into the middle position and leave it disengaged.
- Place the instrument on its side on a cloth. Avoid contact with hot metal surfaces.

Caution: Allow the instrument to cool slowly. Do not use the dispenser again until it has reached room temperature.

The instrument can also be sterilized by the following methods:

- Rinsing with an appropriate liquid, such as alcohol.
- Gas sterilization as long as temperature and pressure do not exceed 121 °C, 15 psi (2 bar).

12 Checking the Instrument Performance

The performance of a Brinkmann Dispenser can be checked using gravimetric testing. This involves the weighing of dispensings of distilled water. The method for gravimetric testing and formulas for calculating systematic error and random error are described below:

- Use a semi- micro balance that has been recently calibrated and located in a vibration-free, draft- free environment.
- All equipment used in the testing process must be isothermal (20 °C constant according ISO 8655-6)
- Set the instrument to the maximum volume.
- Dispense distilled water and then record the weight of the water.
- Perform at least 10 dispensings and weighing operations.

Convert the weights to volumes using the following formula and appropriate density of water for your lab temperature and barometric pressure:

Volume = $\frac{\text{Weight (g)}}{\text{Density of water}}$ The density of water at 16 - 21 °C = 0.998 g/mL 22 - 25 °C = 0.997 g/mL

Calculate the mean (\bar{x}) of the 10 volumes

Mean (\bar{x}) = 1/N $\sum_{i=1}^{N} x_i$

Determine the errors of the instrument by using the formulae below:

Systematic = $\frac{\text{actual volume} - \text{expected volume}}{\text{expected volume}} \times 100 \ [\%]$ Random = $\frac{100s}{\bar{x}} \ [\%]$

s = Standard Deviation actual volume = the mean (\bar{x}) of the 10 volumes expected volume = the maximum setting

 $s = \sqrt{\sum_{\substack{i=1\\N=1}}^{N} (xi - \bar{x})^2}$ N = number of dispensingsxi = adjusted sample weight $\bar{x} = mean$

13 Calibration of the Brinkmann ChemSaver™ Bottletop Dispenser for Liquids with a Density other than Water

The Brinkmann ChemSaver[™] Bottletop Dispenser has been calibrated with water by the manufacturer under the measuring conditions mentioned in section 5. If you are using a liquid that is viscous or has a density significantly different than water, the instrument volume can be fine tuned to compensate for performance differences these types of reagents might cause.

- The instrument and liquid to be dispensed must be equilibrated to the same temperature.
- Set the instrument to the maximum volume.
- Dispense and weigh the maximum volume 10 times.
- Convert the mean value of these weighings to volume using the formula:

Volume = $\frac{\text{Weight}}{\text{Density}}$

- If the volume delivered does not match the volume setting on the dispenser, the fine adjustment knob may be used to increase or decrease the volume dispensed at that setting.
- One full turn of the fine adjustment knob corresponds to the smallest setting increment.
- Turning the knob in the "+" direction increases the volume
- Turning the knob in the "-" direction decreases the volume
- Adjust the fine adjustment knob. Then repeat the steps above to confirm the volume delivered.
- Repeat until desired volume is obtained.

14 Maintenance

The instruments may be partially disassembled for cleaning or replacement of parts. Reference fold out diagram on the cover of this manual and section 17 for available spare parts.

Follow proper safety procedures for hazard prevention. Wear goggles, gloves and protective clothing.

Note: Never use force when disassembling or reassembling!

14.1 Disassembly

Follow the cleaning procedure in section 10 before disassembling the dispenser.

14.1.1 Removing the piston and cylinder sleeve

- The piston (5) may be removed by unscrewing the piston holder (2). Slowly pull the piston (5) out fo the glass cylinder by lifting up on the piston holder (2).
- Insert the appropriate end of the tool (20) into the cylinder ring (19) and remove by turning counter clockwise.
- Remove the cylinder sleeve (3).

14.1.2 Removing the discharge tube sleeve and the discharge tube

Caution: Be careful not to splash the liquid remaining in the discharge tube when removing.

For Brinkmann ChemSaver™ Bottletop Dispenser

- Turn the valve toggle (8) to the recirculate position and remove the toggle by lifting up and off.
- Lift the discharge tube sleeve (9) up and off.
- Pull off the discharge tube (10).

For Brinkmann Bottletop Dispenser

- Lift the discharge tube sleeve (9) up and off.
- Pull off the discharge tube (10) and cap for discharge tube (13).

14.1.3 Removing the filling valve



 The filling valve (17) may be removed by using the tool (20).
 Hold the instrument upside down and place the appropriate end of the tool over the valve. Loosen the valve by turning counterclockwise.

Note: The valve ball is loose inside the valve block. Be careful not to lose it.

Fig. 10

14.2 Cleaning of parts

Parts may be cleaned with a soft brush and appropriate cleaning solution. Do not use abrasive cleaners which could scratch the surface of the piston or cylinder.

Note: Be careful not to damage the seal on the end of the piston.

Allow parts to dry.

14.3 Assembly

- Place the cylinder sleeve (3) over the cylinder.
- Using the appropriate end of the universal tool (20), thread the cylinder ring (19) onto the protective sleeve (7) of the glass cylinder by turning clockwise until it is tight.
- Insert the piston (5) into the cylinder being careful not to damage the piston seal.
- Thread the piston holder (2) together with the cylinder sleeve (3) by turning clockwise until tight.
- Hold the instrument upside down and insert the valve ball (16) into the bottom of the valve block (15).
- Thread the filling valve (17) into the bottom of the valve block (15) by turning clockwise. Use the appropriate end of the tool (20) to tighten the valve.
- Push the discharge tube (10) into the discharge valve (11) or (12).
- For Brinkmann Bottletop Dispensers attach the cap for the discharge tube (13) by guiding the end of the discharge tube (10) through the loop on the cap.
- Mount the discharge tube sleeve (9) onto the valve block housing (14) by first sliding the discharge tube (10) through the bottom of the sleeve, then slide the end of the sleeve over the grooves on the valve block housing.
- If you are assembling a *Brinkmann ChemSaver™ Bottletop Dispenser*, push on the toggle (8) and set to recirculate.

Caution: When reassembling the instrument be sure all parts are securely in place. Test the dispenser for leaks and proper operation with distilled water *before* using the dispenser with reagent.

 After disassembling and reassembling the instrument check the performance of the instrument gravimetrically (section 12).

15 Troubleshooting Guide

Error	Cause	Solution
Piston movement is difficult.	 Cap pushed ont discharge tube. 	o – Remove stopper cap.
	 Formation of cry 	vstals. – Immediately stop dispensing and follow cleaning procedure (sec. 10).
the bolight of	 Damaged pistor 	 Immediately stop dispensing and follow cleaning procedure (sec.10), replace piston or send the dispenser to Brinkmann Instruments for repair if necessary.
No reagent is aspirated.	 Adjusted to mini volume setting. 	mum – Use volume setting knob to set higher volume.
1279 (2019 (2017) 	 Filling valve clog or sticking. 	ged – Follow cleaning procedure. Remove filling valve (sec. 10), replace valve if necessary.
Air bubbles in aspirated liquid.	 Instrument not properly primed. 	 Follow the procedure for removing air from the instrument (sec.9).
	 Filling tube not attached correct or damaged. 	 Follow cleaning procedure (sec.10). Push filling tube all the way up onto the filling valve. Shorten extend or replace filling tube when necessary.
	 Reagent aspirate too quickly. 	ed – Raise piston slower when aspirating.
	 Filling valve loos or damaged. 	e – Follow cleaning procedure (sec 10). Remove filling tube. Tighten valve or replace.
	 End of filling tube above liquid leve 	e is – Extend filling tube so the end is below the liquid level.
		 Fill empty bottle.
Dispensed volume too low.	 Filling valve loose or damaged. 	 Follow cleaning procedure (sec 10). Remove filling tube. Tighten valve or replace.
	 Filling tube not attached correctl or damaged. 	 Follow cleaning procedure (sec.10). Push filling tube all the way up onto the filling valve. Shorten extend or replace filling tube when necessary.

16 Sending in for Repair

If a problem cannot be solved with the aid of this instruction manual, we recommend you return the Brinkmann Bottletop Dispenser to Brinkmann Instruments for repair.

The dispenser must be cleaned and decontaminated before returning to Brinkmann Instruments. Never send an instrument filled with reagent. Returned instruments must be free from toxic or biohazardous material.

Instruments from biological applications must be steam sterilized.

The application of force in opening or disassembling of the instrument will void the warranty.

Send the cleaned instrument to Brinkmann Instrument Services, Inc. at the address printed on the back of this manual.

In your package include information describing the observed fault and the liquids which have been dispensed. For warranty claims include a copy of an invoice or purchase order as proof of purchase.

17 Ordering Information

17.1 Bottletop Dispensers

Brinkmann ChemSaver™ Bottletop Dispensers

Adjustable volume

olume Range (mL)	Thread	Cat No
0.1 - 2.5	32 mm	22 22 090-0
1 – 5	32 mm	22 22 100-1
2 - 10	32 mm	22 22 110-8
5 - 25	45 mm	22 22 120-5
10 - 50	45 mm	22 22 130-2
20 - 100	45 mm	22 22 140-0

Brinkmann Bottletop Dispensers

Adjustable volume

/olume Range (mL)	Thread	
0.1 - 2.5	32 mm	22 22 000-4
1 – 5	32 mm	22 22 010-1
2 - 10	32 mm	22 22 020-9
5 – 25	45 mm	22 22 030-6
10 - 50	45 mm	22 22 040-3
20 - 100	45 mm	22 22 050-1

Fixed volume Volume (mL) 1 5 10	Thread 32mm 32mm 45mm	Cat.No. 22 22 060-8 22 22 070-5 22 22 080-2
17.2 Accessories Extended Discharge Tube For Dispenser Sizes 1 to10 mL22 22 300-3 25 to100 mL22 22 305-4	9, 80 cm long 1	
<i>Threaded adapter,</i> polypro <i>for 1 – 10 mL</i> 28 mm 38 mm 45 mm set one of each size 28 mm	pylene, set of 3 , 38 mm, 45mm	22 22 310-1 22 22 315-1 22 22 320-8 22 22 335-6
for 25 – 100 mL 33 mm 38 mm 40 mm		22 22 312-7 22 22 318-6 22 22 322-4
<i>Standard tapered joint ada</i> STJ 24/40 STJ 29/42	apters, polypropylene, pkg. of	1 22 22 325-9 22 22 330-5
<i>Threaded adapter,</i> Teflon, p <i>for 1 – 10 mL</i> 28 mm 38 mm 45 mm	skg. of 1	22 22 340-2 22 22 345-3 22 22 350-0

38 mm 40 mm

for 25 - 100 mL inner diameter? 33 mm afthecap

Adapter for Drum 45 mm / 2.25 inch

22 22 360-7

22 22 352-6 22 22 355-1 22 22 357-7

\$ 27.00

17.3 Spare Parts

For Brinkmann ChemSaver™ Bottletop Dispensers

Piston (5) complete with piston holder (2) and fine adjustment (1)

Cat.No. 0.1 - 2.5 mL 22 22 500-6 1 -5 mL 22 22 505-7 2 10 ml 22 22 510-3 - 25 5 mL 22 22 515-4 10 - 50ml 22 22 520-1 20 - 100mL 22 22 525-1 Cylinder sleeve (3) 0.1 - 2.5 mL 22 22 600-2 1 -5 ml 22 22 605-3 2 - 10 mL22 22 610-0 5 - 25 ml 22 22 615-1 10 – 50 mL 22 22 620-7 20 – 100 ml 22 22 625-8 Discharge tube (10) 0.1 - 2.5 mL, 1 - 5 mL, 2 - 10 mL 22 22 560-0 5 - 25 mL, 10 - 50 mL, 20 - 100 mL 22 22 565-1 Discharge tube sleeve (9) 0.1 - 2.5 mL, 1 - 5 mL, 2 - 10 mL 22 22 550-2 5 - 25 mL, 10 - 50 mL, 20 - 100 mL 22 22 555-3 Discharge valve toggle (8) 0.1 - 2.5 mL, 1 - 5 mL, 2 - 10 mL 22 22 530-8 5 - 25 mL, 10 - 50 mL, 20 - 100 mL 22 22 535-9 For Brinkmann Bottletop Dispensers Piston (5) complete with piston holder (2) 0.1 -2.5 mL 22 22 700-9 1 - 5 mL22 22 705-0 2 - 10 mL22 22 710-6 5 - 25 mL 22 22 715-7 10 - 50 mL 22 22 720-3 20 - 100mL 22 22 725-4 1 mL fixed 22 22 730-1 5 mL fixed 22 22 735-1 10 mL fixed 22 22 740-8

<i>Discharge tube</i> (10) 0.1 – 2.5 mL, 1 – 5 mL, 2 – 10 mL 5 – 25 mL, 10 – 50 mL, 20 – 100 mL	Cat.No. 22 22 765-3 22 22 565-1
Cylinder sleeve (3) 0.1 - 2.5 mL 1 - 5 mL 2 - 10 mL 5 - 25 mL 10 - 50 mL 20 - 100 mL 1 mL fixed 5 mL fixed 10 mL fixed	22 22 815-3 22 22 820-0 22 22 825-1 22 22 830-7 22 22 835-8 22 22 840-4 22 22 845-5 22 22 850-1 22 22 855-2
<i>Discharge tube sleeve</i> (9) 0.1 - 2.5 mL, 1 - 5 mL, 2 - 10 mL 5 - 25 mL, 10 - 50 mL, 20 - 100 mL	22 22 755-6 22 22 760-2
For both Model Dispensers Filling Valve (17)	
0.1 – 2.5 mL, 1 – 5 mL, 2 – 10 mL 5 – 25 mL, 10 – 50 mL, 20 – 100 mL	22 22 765-3 22 22 765-1
<i>valve ball</i> (16) 0.1 – 2.5 mL, 1 – 5 mL, 2 – 10 mL 5 – 25 mL, 10 – 50 mL, 20 – 100 mL	22 22 660-6 22 22 665-7
<i>Telescopic filling tube</i> <i>Drying tube</i> , cpl. (not filled) <i>Tool</i> <i>Enlarging ring f. Tool</i> (for 100 mL)	22 22 675-4 27 31 110-5 22 22 680-1 22 22 682-7

Quality assurance

This Brinkmann ChemSaver[™] Bottletop Dispenser has been manufactured and tested under stringent quality controls to ensure its accuracy and precision. It is guaranteed against defects in workmanship or faulty parts

for 12 months

after the date of delivery. Please refer to the notes in this instruction manual. If, in spite of our extensive controls, the Brinkmann ChemSaver™ Bottletop Dispenser is defective, please return it to Brinkmann Instruments.

图

X

BOTTLETOP DISPENSER

Serial No.	91544		10-50 mi
. 50 ml c	d = -0,02 %	cv =	0,08 %
d = devia	tion / Unrich	itigkeit	
cv = coeffi	cient of varia	ation / L	Jnpräzision
Acc. to / na	ach DIN 126	50	
Final chec	k / Endkontre	olle	

× ·

Brinkmann Instruments, Inc. One Cantiague Road, TO. Box 1019. Westbury, New York 11590-0207 (USA) Phone: 800-645-3050 Fax: 516-334-7506 e-mail: info@brinkmann.com Internet: www.brinkmann.com

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eppendorf

SHEL LAB

CONSTANT TEMPERATURE OVEN MICROPROCESSOR CONTROLLED

> FLOOR MODELS: FX14-2 & FX28-2

INSTALLATION AND OPERATIONAL MANUAL

Sheldon Manufacturing Inc. P.O. Box 627 Cornelius, Oregon 97113EMAIL: tech@Shellab.comINTERNET: http://www.Shellab.com/~Shellab1-800-322-4897(503) 640-3000FAX (503) 640-1366

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- MAINTENANCE
 - TROUBLESHOOTING
 - PARTS LIST
 - UNIT SPECIFICATIONS

SCHEMATICS

REV. 01/08 4861581-2

These units are general purpose air ovens for professional, industrial or educational use where the preparation or testing of materials is done at approximately atmospheric pressure and no flammable, volatile or combustible materials are being heated. These units are not intended for hazardous or household locations or use.

Section

RECEIVING AND INSPECTION

Your satisfaction and safety require a complete understanding of this unit. Read the instructions thoroughly and be sure all operators are given adequate training before attempting to put the unit in service. NOTE: This equipment must be used only for its intended application; any alterations or modifications will void your warranty.

- **1.1 Inspection:** The carrier, when accepting shipment, also accepts responsibility for safe delivery and is liable for loss or damage. On delivery, inspect for visible exterior damage, note and describe on the freight bill any damage found, and enter your claim on the form supplied by the carrier.
- **1.2** Inspect for concealed loss or damage on the unit itself, both interior and exterior. If necessary, the carrier will arrange for official inspection to substantiate your claim.
- **1.3 Return Shipment:** Save the shipping crate until you are sure all is well. If for any reason you must return the unit, first contact your customer service representative for authorization. Supply nameplate data, including model number and serial number.
- **1.4 Accessories:** Verify that all of the equipment indicated on the packing slip is included with the unit. Carefully check all packaging before discarding. The HX14-2 are equipped with 3 shelves, 12 shelf clips and 4 leveling feet. The FX28-2 is equipped with 6 shelves, 24 shelf clips and 4 leveling feet.



GRAPHIC SYMBOLS

Your oven is provided with a display of graphic symbols which should help in identifying the use and function of the available user adjustable components.



Section

INSTALLATION

Local city, county or other ordinances may govern the use of this equipment. If you have any questions about local requirements, please contact the appropriate local agency. Installation may be performed by the end user. Under normal circumstances this unit is intended for use indoors, at room temperatures between 5° and 40°C, at no greater than 80% Relative Humidity (at 25°C) and with a supply voltage that does not vary by more than 10%. Customer service should be contacted for operating conditions outside of these limits.

Installation of the FX28-2 requires hard wiring and should be performed by a qualified electrical technician. The next higher circuit breaker value above the data plate amperage may be used provided the requirements in article 422 of the National Electric Code are met (USA). The FX14-2 can be installed by the end user without a technician.

- **3.1 Power Source:** The electrical supply circuit to the oven must conform to all national and local electrical codes. Consult the incubator's serial data plate for the voltage, cycle wattage and ampere requirements before making connection. VOLTAGE SHOULD NOT VARY MORE THAN 10% FROM THE SERIAL PLATE RATING. This unit is intended for 50/60 Hz application. A separate circuit is recommended to prevent possible loss of product due to overloading or failure of other equipment on the same circuit.
- **3.2 Location:** When selecting a site for the oven, consider all conditions which may affect performance, such as extreme heat from steam radiators, stoves, ovens autoclaves, etc. Avoid direct sun, fast-moving air currents, heating/cooling ducts, and high traffic areas. To ensure air circulation around the unit allow a minimum of 30 cm between the unit and any walls or partitions which might obstruct free airflow.
- **3.3 Lifting / Handling:** These units are heavy and care should be taken to use appropriate lifting devices that are sufficiently rated for these loads. Units should only be lifted from their bottom surfaces. Doors, handles and knobs are not adequate for lifting or stabilization. The unit should be completely restrained from tipping during lifting or transport. All moving parts, such as shelves and trays should be removed and doors need to be positively locked in the closed position during transfer to prevent shifting and damage.
- **3.4 Leveling:** The unit must sit level and solidly. Leveling feet are supplied and must be installed in the four holes in the bottom corners of the unit. With the unit

standing upright, turn the leveling feet counterclockwise to raise level. If the unit must be moved, turn the leveling feet in all the way to prevent damage.

3.5 Cleaning: The oven interior was cleaned at the factory, but not sterilized. Remove all interior parts if assembled and clean with a disinfectant that is appropriate to your application. DO NOT USE chlorine-based bleaches or abrasives as this will damage the stainless steel interior. DO NOT USE spray cleaners that might leak through openings and cracks and get on electrical parts or that may contain solvents that will harm the coatings. A similar periodic cleaning is recommended.

WARNING: Never clean the unit with alcohol or flammable cleaners with the unit connected to the electrical supply. Always disconnect the unit form the electrical service when cleaning and assure all volatile or flammable cleaners are evaporated and dry before reattaching the unit to the power supply.

3.5 Burn In: It is recommended that the unit go through a "burning in" process prior to operation. This is to eliminate the smoking of protective coatings on the element. Read sections 4, 5 and 6 carefully to understand operating requirements. To burn in turn the Overtemperature Safety to maximum and set the digital display to 200°. Run a minimum of one (1) hour until smoke dissipates.

Section

PRECAUTIONS

- **4.1** These units have been designed with dampered vents from the chamber. In order to work effectively and safely, some precautions will need to be taken by the operator.
 - **A.** In most applications, the EXHAUST damper will need to be opened during drying or degassing for best results.
 - B. THIS OVEN IS NOT DESIGNED TO HANDLE COMBUSTIBLE GASSES AND IS NOT AN EXPLOSION PROOF UNIT. Do not place explosive, combustible, or flammable materials into the chamber.
 - **C.** Some of the outgassed by-products may be hazardous or unpleasant to operating personnel. If this is the case, the exhausts should be positively ventilated to the outside and dealt with according to local regulations.
- **4.2** Do not operate near noxious fumes.
- **4.3** Do not place sealed or filled containers in the oven chamber.
- **4.4** Do not cut or remove the ground prong from the power cord.
- **4.5** Be sure that the power supply is of the same voltage as specified.
- **4.6** Disconnect the unit from the electrical source before proceeding to make any electrical repairs or replacements.
- **4.7** If a mercury thermometer is used and breakage should occur, all spilled mercury MUST be completely removed from the chamber before continuing operation.
- **4.8** This oven is NOT designed for use in Class I, II, or III locations as defined by the National Electrical Code.
- **4.9** This oven is not intended, nor can it be used, as a patient connected device.



CONTROL PANEL OVERVIEW



- A. Power Switch
- B. Timer Switch
- C. Overtemperature Safety Light
- D. Overtemperature Safety thermostat
- E. Timer Control
- F. Main Temperature Controller
- G. Temperature Activated Light
- **5.1 Power Switch:** The main power switch on the control panel (green lighted I/O) controls all power to the unit and must be in the I/ON position before any systems are operational. The switch will be lighted when in the I/ON position.
- **5.2 Timer Switch:** The black I/O switch marked TIMER is located to the right of the power switch. It controls the power to the time circuit. In the off position the oven heat is controlled with no timed duration. In the on position the heat is controlled for a timed interval and then the heat shuts off.
- **5.3 Main Temperature Control:** This control is marked SET/TEMPERATURE and consists of the digital display and UP/DOWN arrow pads for inputting set point temperatures and calibration.

- **5.4 Overtemperature Safety Thermostat:** This control is marked HIGH LIMIT and is equipped with a graduated dial from 0-10. It is independent of the Main Controller and guards against any failure which would allow the temperature to rise past the Main Controllers set point. This allows continued operation of the oven until the problem can be corrected or service can be arranged. It is not recommended that the unit be operated for extended periods of time using only the Overtemperature Safety as the controller as temperature uniformity will suffer.
- **5.5 OTP Light:** This pilot lamp is marked OTP and is directly above the Overtemperature Safety Thermostat. The light will come on when the Safety Thermostat has been activated and taken control of the oven. Under normal operating conditions this pilot lamp should never be on.
- **5.6 Timer Control:** This control is marked SET/TIMER and consists of a digital display, UP/DOWN arrow pads, a RESET "PUSH" pad, a START/STOP "PUSH" pad and a TIMER ACTIVATED Light. This control provides the ability to set a timed heat interval, activate the start-up of the timed heat cycle and shut down the timed heat cycle automatically.
- **5.7 Temperature Activated Light:** This pilot lamp will be lit when the elements are receiving power.

Section

OPERATION

- **6.1 Connection to Power Supply:** Assure that the electrical power supply is properly configured and rated for the oven. The FX28-2 must be hard wired as stated in Section 3.0, Installation.
- **6.2** Push the POWER switch to the ON position. The digital temperature display will indicate a temperature value. Turn the Overtemperature Safety Thermostat to its maximum position, clockwise, by using a flat-head screwdriver.
- **6.3** Set Main Temperature Controller: Enter desired set point temperature. To enter set point mode on the controller, press either the Up or Down arrow pad one time. The digital display will start to blink, going from bright to dim. While blinking, the digital display is showing the set point. To change the set point, use the Up and Down arrow pads. If the arrow pads are not pressed for five (5) seconds, the display will stop blinking and will read the temperature of the unit. Note that the High Limit Thermostat should be turned to its maximum position until the unit has stabilized at desired set point temperature. Allow the oven at least 24 hours to stabilize.
- **6.4 Calibration:** Temperature calibration is done once the unit is installed in its working environment and has been stabile at set point for several hours. Place a certified reference thermometer in the chamber by either placing it directly inside, or through the access tube at the top left of the unit. Be certain the thermometer is not touching any shelving. Allow the temperature to stabilize again until the thermometer reads a constant value for one hour. Compare the digital display with the reference thermometer. If there is an unacceptable difference, put the display into calibration mode by pressing both the Up and Down arrow pads at the same time until the two outside decimal points begin to flash. While the decimal points are flashing the display reads the correct value. Allow the oven temperature to stabilize again, and recalibrate if necessary.

NOTE: Temperature accuracy should be checked at least monthly or after the unit has been turned off for an extended period of time.

6.5 Set Overtemperature Safety Thermostat: As mentioned in step 6.3, the Safety Thermostat should be initially set to its maximum position to allow the unit to stabilize. Once the oven is stabile at the desired set point, turn the Safety

Thermostat counterclockwise with a flat-head screwdriver until the OTP light turns ON. Next, turn the Thermostat clockwise just until the OTP light turns OFF. Then turn the thermostat clockwise two (2) of the smallest divisions on its scale past the point where the light went out. This sets the Safety Thermostat at a temperature approximately 10°C above the Main Temperature set point. Note that the Safety is in series with the output from the control relay, and the OTP light will be blinking when the Main Temperature Controller is calling for heat.

- **6.6 Set Timer Display:** Turn the Timer switch to the ON position. The SET/TIMER display digits will light with no lighted decimals showing (See Figure 2). Note that if during any of the following steps, several seconds elapse with no arrow pad or RESET pad activity, the timer will default to the present displayed setting and it will be necessary to restart all functions over again. The values must be programmed in a consecutive manner with no delays between settings or the default will occur.
 - A. Hour Function: Press and hold the RESET pad until the digits start blinking and a blinking decimal point is between digits 2 and 3. In this mode, pressing the UP or DOWN arrow pads increases or decreases the whole hour value from 0 to 99 (digits 1 and 2).
 - **B. Ten Minute Function:** After the correct value for hours is set, push the RESET pad again. The blinking decimal will now move one digit to the right between digits 3 and 4. Pushing the UP or DOWN arrow pads will increase or decrease the ten minute function allowing values between 0 to 5 to be set (digit 3).
 - **C. One Minute Function:** After the correct ten minute value is set, push the RESET pad again. The blinking decimal point will now move one digit to the right beyond digit 4 and be located at the extreme bottom right of the display. With the display in this mode, pushing the UP or DOWN arrow pad will increase the one minute function allowing the value of digit 4 to be adjusted between 0 and 9.
 - **D.** Activation: Pause until timer stops blinking. After all settings are made, push the START/STOP button. The timer activated light will come on and after a brief pause, the present oven temperature settings will be valid and heating will begin. The oven will now heat up, control at the set point and stop after the timed period on the SET/TIMER display has elapsed.

Note that when the system is in the timer mode, the heating circuit is deenergized until the START/STOP button is pushed or the TIMER switch is turned Off. If a time change or correction is necessary and the timer has already been activated, push the START/STOP button to "STOP" the timer, then repeat steps A through D above.

6.7 To set the timer so that timed operation will not start until the oven is stabile at set point, pre-heat the oven in the normal mode until the desired temperature has stabilized. Turn on the TIMER switch. Push and hold the RESET button until the timer display blinks, this is to be sure that the pre-set timed value is correct. Press the START/STOP button to activate the timer.



Figure Two



Section

MAINTENANCE

7.1 Cleaning: Clean the oven interior and remove and clean shelves on a regular basis. Use a disinfectant that is suitable for your application. DO NOT USE chlorine-based bleaches or abrasives, as this will damage the stainless steel interior. DO NOT USE spray cleaners that might leak through openings and cracks and get on electrical parts or that may contain solvents that will harm the coatings. A similar periodic cleaning is recommended. Use care when cleaning the door gasket to prevent damage which could impair the positive door seal.

WARNING: Never clean the unit with alcohol or flammable cleaners with the unit connected to the electrical supply. Always disconnect the unit form the electrical service when cleaning and assure all volatile or flammable cleaners are evaporated and dry before reattaching the unit to the power supply.

- **7.2 Storage:** To prepare the unit for storage, remove all shelves and shelf clips, dry the chamber completely and disconnect the power supply. Be certain that the door is positively locked in the closed position. See Section 3.3, Lifting/Handling, for proper transport procedures.
- **7.3** No maintenance is required on the electrical components. If the unit fails to operate as specified, please see the Troubleshooting guide, Section 8.0, before calling for service.

Section

TROUBLESHOOTING

TEMPERATURE

Temperature too high.	
	 Controller set too high-see section 6.3 Controller failed on – call Customer Service. Wiring error – call Customer Service.
Display reads "HI" or "400"+.	
	Probe is unplugged, is broken or wire to sensor is broken – trace wire from display to probe; move wire and watch display to see intermittent problems
Chamber temp spikes over set point and then settles to set point.	
	Recalibrate – see section 6.4.
Temperature too low	
	 1/ High limit set too low – see section 6.5. 2/ Controller set too low – see section 6.3. 3/ Unit not recovered from door opening – wait for display to stop changing. 4/ Unit not recovered from power failure or being turned off – ovens will need several hours to warm up and stabilize. 5/ Element failure – compare current draw to data plate. 6/ Controller failure – call Customer Service. 7/ High limit failure – confirm with front panel lights that Safety Thermostat is operating correctly. 8/ Wiring problem – check all functions and compare wiring to schematic in section 9.0 - especially around any areas recently worked on. 9/ Loose connection – check control panel for loose connections.
Display reads "LO"	
	 Bad probe or disconnected – call Customer Service. If ambient temperature is lower than range of unit – compare set points and ambient temperature to rated specifications in section 9.0.
Unit will not heat over a temperature that is below set point	
	 1/ Confirm that fan is moving and that amperage and voltage match data plate – check for air movement in chamber. 2/ Confirm that set point is set high enough –turn Safety

	 Thermostat all the way clockwise and see if OTP light comes on. 3/ Check connections to sensor. 4/ Check calibration – using independent thermometer, follow instructions in section 6.4.
Unit will not heat up at all	
	 1/ Check amperage – amperage should be virtually at maximum rated (data plate) amperage. 2/ Do all controller functions work? 3/ Is the Safety Thermostat set high enough? – for diagnostics, should be fully clockwise with the OTP light never on. 4/ Has the fuse/circuit breaker blown? 5/ Has timer turned unit off?
Indicated chamber temperature unstable	
	 1/±0.1 may be normal. 2/ Is fan working? -verify movement of air in chamber. 3/ Is ambient room temperature radically changing – either door opening or room airflow from heaters or air conditioning ? – stabilize ambient conditions. 4/ This may happen if exhaust stack is 100% open or if power exhaust is cycling – adjust stack to at least ¼ closed. 5/ Sensor miss-located, damaged or wires may be damaged - check mounts for control and OTP sensors, then trace wires or tubing between sensors and controls. 6/ Calibration sensitivity – call Customer Service. 7/ High limit set too low – be sure that Safety Thermostat is set more than 5 degrees over Main Controller set point; check if OTP pilot is on continuously; turn controller knob completely clockwise to see if problem solved then follow instructions in section 6.5 for correct setting. 8/ Electrical noise – remove nearby sources of RFI including motors, arcing relays or radio transmitters 9/ Bad connection on temperature sensor or faulty sensor – check connectors for continuity and mechanical soundness while watching display for erratic behavior; check sensor and wiring for mechanical damage. 10/ Bad connections or faulty solid state relay – check connectors for mechanical soundness and look for corrosion around terminals or signs of arcing or other visible deterioration. 11/ If set point is below 60 degrees, temperature can be unstable. See unit specifications for individual ranges.
Will not maintain set point	
	 Assure that set point is at least 5 degrees over ambient. See if ambient is fluctuating; check for adjacent open doors or HVAC duct openings – stabilize ambient conditions.
Display and reference	

thermometer don't match	
	 Calibration error – see section 6.4. Temperature sensor failure – call Customer Service. Controller failure – call Customer Service. Allow at least two hours to stabilize. Verify that reference thermometer is certified.
Can't adjust set points or calibration	
	 1/ Turn entire unit off and on to reset. 2/ If repeatedly happens, call Customer Service.
Calibrated at one temperature, but not at another	
	This can be a normal condition when operating temperature varies widely. For maximum accuracy, calibration should be done at or as close to the set point temperature.
N	IECHANICAL
Motor doesn't move	
	 If shaft spins freely: check connections to motor and check voltage to motor. If shaft rubs or is frozen, relieve binding and retest.
Motor makes noise	
	 Make sure that the fan or blower wheel is not contacting its housing. Adjust the motor mounting bracket position to re-center the fan or blower wheel, if necessary. Check the fan or blower wheel for damage or out of balance condition. Replace the fan or blower wheel if it is damaged or out of balance. Turn the motor shaft to make sure that it spins freely. If it binds or the bearings make a rubbing or scrapping sound then replace the motor.
Door not sealing	
	 Adjust hinge blocks or twist the door. Confirm that unit has not been damaged and body is not out of square. Check physical condition of gasket for tears or punctures.
	OTHER
Controller on at all times - "locked-up"	
	 Adjust set point to room temperature. If the unit is still heating, replace the solid state relay. Z/Turn unit off and on to reset. If cannot change any condition on the front panel, call Customer Service.
Controller timer resets on its own	
	 Confirm that power from wall is consistent and within specifications. Call Customer Service with serial number.
Front panel displays are all off	
---	--
	 1/ Check connections to the temperature display control board and assure that all are tight and in the correct orientation. 2/ Check for wire damage.
Unit or wall fuse/circuit breaker is blown	
	 1/ Check wall power source. 2/ Compare current draw and compare to specs on data plate. 3/ See what other loads are on the wall circuit.
Unit will not turn on	
	 Check wall power source. Check fuse/circuit breaker on unit or in wall. See if unit is on, e.g., fan or heater, and just controller is off. Check all wiring connections, especially around the on/off switch.
Unit is smoking – Out of box	
	This is not an uncommon occurrence when first operating new units. Put unit under vent and run at high temperature for one hour until smoke dissipates.
Contamination in chamber	
	 See cleaning procedure in section 7.0. Develop and follow standard operating procedure for specific application; include definition of cleaning technique and maintenance schedule.
Contamination in sample	
	 See "Contamination in chamber". Reduce air flow in chamber by dampening down exhaust port; be sure to verify adequate temperature uniformity at the reduced air flow. Protect open samples from areas of maximum air current, e.g., inlet air ducts.

Section

PARTS LIST

Description	220V
Adjustable Feet	2700506
Black I/O Switch, Timer	X1000124
Blower Motor	4880548
Cordset, FX14-2	1800500
Element FX14-2	9570737
Element FX28-2	9570730P
EMI Filter FX14-2	2800502
EMI Filter FX28-2	2800504
Fuse, 10amp 5 x 20mm	3300516
Green I/O Switch, Power	103351
Knob, OTP	4450506
Pilot Light, Heating	200021
Pilot Light, OTP	200020
Safety Thermostat	1750615
Shelf Clip	200137
Shelf FX14-2 & FX28-2	5130581
Temp/Time Control	1750613

UNIT SPECIFICATIONS

These units are 240 volt. Please refer to the unit data plate for its individual specifications.

Unit	Dimensions WxDxH		Weight		Capacity
	Exterior	Interior	Shipping	Net	Cubic Ft
FX14-2	37X34X47	30.75x24.75x47	490 lbs.	280 lbs.	14
FX28-2	37.5x24.75x78.25	30.75x24.75x62.5	550 lbs.	390 lbs.	28

Temp. °C	Range	Uniformity	Heat-up Time to 150°	Recovery to 150°	Sensitivity
FX14-2	Amb. +5 to 220°C	<u>+</u> 1.5° @ 100°	14 min.	4 min (door open 30 sec.)	<u>+</u> .05°
FX28-2	Amb. +5 to 220°C	<u>+</u> 1.5° @ 100°	14 min.	4 min (door open 30 sec.)	<u>+</u> .05°

WIRE DIAGRAM FX14-2



9850511 1375FM, L3B0FM, SL-09, SL-D14



SHELDON MANUFACTURING, INC. LIMITED WARRANTY (Parts only, exclusive of labor)

Sheldon Manufacturing, Inc., ("Manufacturer") warrants for the original user of this product that all parts, not including finished goods products, it manufactures or resells will be free from defects in material and workmanship for a period of one year from the date of delivery of this product to the original user (the "Warranty Period"). During the Warranty Period, Manufacturer, at its election and expense, will repair or replace parts that are proven to Manufacturer's satisfaction to be defective, or, at Manufacturer's option, refund the price or credit the price of any parts that are proven to Manufacturer's satisfaction to be defective. This warranty does not cover any labor or damage due to accident, misuse, negligence, or abnormal use. Use of parts in a system that includes components not manufactured by Manufacturer is not covered by this warranty. Any alteration or removal of the serial number on Manufacturer's parts will void this warranty are governed by the laws of the state of Oregon without regards to the principles of conflicts of laws thereof. If any provision of this limited warranty is held to be unenforceable by any court of competent jurisdiction, the remainder of this limited warranty will remain in full force and effect.

This warranty is in lieu of and excludes all other warranties or obligations, either express or implied. Manufacturer expressly disclaims all implied warranties, including without limitation, the warranties of merchantability and fitness for a particular purpose.

...........

For fast and efficient support, please have the following information available anytime you request service:

Model _____ Serial No. _____ Part No. _____



Type F62700 Furnace OPERATION MANUAL AND PARTS LIST Series 1276

Model No.	Voltage	Control	Display	
F62730	220-240V	Single Set Point	°C	
F62730-80	220-240V	Programmable	°C	
F62730-33	220-240V	Single Set Point	°C	
F62730-33-80	220-240V	Programmable	°C	
F62734	100V	Single Set Point	°C	
F62735	120V	Single Set Point	°C	
F62735-80	120V	Programmable	°C	

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Safety Information

Alert Signals



Warning

Warnings alert you to a possibility of personal injury.



Caution

Cautions alert you to a possibility of damage to the equipment.



Note

Notes alert you to pertinent facts and conditions.



Hot Surface

Hot surfaces alert you to a possibility of personal injury if you come in contact with a surface during use or for a period of time after use. This manual contains important operating and safety information. The user must carefully read and understand the contents of this manual prior to the use of this equipment.

Your Barnstead Thermolyne furnace has been designed with function, reliability, and safety in mind. It is the user's responsibility to install it in conformance with local codes. For safe operation, please pay attention to the alert signals throughout the manual.

Warnings

To avoid electrical shock, this furnace must:

- 1. Use a properly grounded electrical outlet of correct voltage and current handling capacity.
- 2. Disconnect from the power supply prior to maintenance and servicing.
- 3. Have the door switch operating properly.

To avoid burns, this furnace must:

Not to be touched on the exterior or interior surfaces during use or for a period of time after use.

To avoid personal injury:

- Do not use in the presence of flammable or combustible materials; fire or explosion may result. This device contains components which may ignite such materials.
- 2. Refer servicing to qualified personnel.

Please note the following WARNINGS:

WARNING

This warning is presented for compliance with California Proposition 65 and other regulatory agencies and only applies to the insulation in this product. This product contains refractory ceramic, refractory ceramic fiber or fiberglass insulation, which can produce respirable dust or fibers during disassembly. Dust or fibers can cause irritation and can aggravate preexisting respiratory diseases. Refractory ceramic and refractory ceramic fibers (after reaching 1000°C) contain crystalline silica, which can cause lung damage (silicosis). The International Agency for Research on Cancer (IARC) has classified refractory ceramic fiber and fiberglass as possibly carcinogenic (Group 2B), and crystalline silica as carcinogenic to humans (Group 1).

The insulating materials can be located in the door, the hearth collar, in the chamber of the product or under the hot plate top. Tests performed by the manufacturer indicate that there is no risk of exposure to dust or respirable fibers resulting from operation of this product under normal conditions. However, there may be a risk of exposure to respirable dust or fibers when repairing or maintaining the insulating materials, or when otherwise disturbing them in a manner which causes release of dust or fibers. By using proper handling procedures and protective equipment you can work safely with these insulating materials and minimize any exposure. Refer to the appropriate Material Safety Data Sheets (MSDS) for information regarding proper handling and recommended protective equipment. For additional MSDS copies, or additional information concerning the handling of refractory ceramic products, please contact the Customer Service Department at Barnstead International at 1-800-553-0039.

Introduction

Intended Use

The Type F62700 furnace is a general laboratory and heat treating furnace. For optimum element life, it is suggested that this furnace be used for applications requiring temperatures from 400°F (204°C) to 1600°F (871°C) for continuous use, or temperatures from 1600°F (871°C) to 1832°F (1000°C) for intermittent use. Continuous use is operating the furnace for more than 3 hours and intermittent use is operating the furnace for less than 3 hours.

The unit consists of: 1) muffle heating chamber, 2) a microprocessor control and 3) a door interlock relay for user safety.

General Usage

Do not use this product for anything other than its intended usage.

Principles of Operation

The furnace chamber incorporates a heating element that is embedded into the side walls and top and back of heating chamber. The ceramic fiber oval muffle with four heating surfaces creates even heat distribution within the chamber. The temperature is controlled by a microprocessor control with a type K Chromel/Alumel Thermocouple. A fan is provided in the furnace to provide forced air cooling for the temperature controller. A door safety switch removes power to the heating elements whenever the furnace door is opened.

General Specifications

MODEL NUMBER		F62730, F62730-80	F62730-33 F62730-33-80	F62734	F62735, F62735-80
OVERALL	WIDTH	23-1/2 (59.7 CM)	23-1/2 (59.7 CM)	23-1/2 (59.7 CM)	23-1/2 (59.7 CM)
DIMENSIONS	HEIGHT	16-1/4 (41.3 CM)	16-1/4 (41.3 CM)	16-1/4 (41.3 CM)	16-1/4 (41.3 CM)
IN. (CM)	DEPTH	18-3/4 (47.6 CM)	18-3/4 (47.6 CM)	18-3/4 (47.6 CM)	18-3/4 (47.6 CM)
CHAMBER	WIDTH	11-1/2 (29.2 CM)	11-1/2 (29.2 CM)	11-1/2 (29.2 CM)	11-1/2 (29.2 CM)
DIMENSIONS	HEIGHT	7-1/2 (19.1 CM)	7-1/2 (19.1 CM)	7-1/2 19.1 CM)	7-1/2 19.1 CM)
IN. (CM.)	DEPTH	11-1/2 (29.2 CM)	11-1/2 (29.2 CM)	11-1/2 (29.2 CM)	11-1/2 (29.2 CM)
WEIGHT	LBS. (KG)	55 LBS (25 KG)	55 LBS (25 KG)	55 LBS (25 KG)	55 LBS (25 KG)
ELECTRICAL	VOLTS	220-240	220-240	100	120
RATINGS	AMPS	6.2	6.2	14.8	12.4
	WATTS	1488	1488	1488	1488
	FREQ. PHASE	50/60 1	50/60 1	50/60 1	50/60 1
MAXIMUM OPERATING	INTERMITTENT	1832°F (1000°C)	1832°F (1000°C)	1832°F (1000°C)	1832°F (1000°C)
	CONTINUOUS	1600°F (871°C)	1600°F (871°C)	1600°F (871°C)	1600°F (871°C)

Environmental Conditions

Operating: 17°C - 27°C; 20% to 80% relative humidity, non-condensing. Installation Category II (over-voltage) in accordance with IEC 664. Pollution Degree 2 in accordance with IEC 664. **Altitude limit:** 2,000 meters.

Storage: -25°C to 65°C; 20% to 80% relative humidity.

Declaration of Conformity

(for -33 models only)

Barnstead International hereby declares under its sole responsibility that this product conforms with the technical requirements of the following standards:

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ory equipment for
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per the provisions of the Electromagnetic Compatibility Directive 89/336/EEC, as amended by 92/31/EEC and 93/68/EEC, and per the provisions of the Low Voltage Directive 73/23/EEC, as amended by 93/68/EEC.

The authorized representative located within the European Community is:

Electrothermal Engineering Ltd. 419 Sutton Road Southend On Sea Essex SS2 5PH United Kingdom

Copies of the Declaration of Conformity are available upon request.

Unpacking

Visually check for any physical damage to the shipping container. Inspect the equipment surfaces that are adjacent to any damaged area. Open the furnace door and remove packing material from inside the furnace chamber. Vacuum the chamber prior to use to remove the insulation dust due to shipping.

The furnace is supplied with two hearth plates. Place the hearth plates on the bottom of the chamber.

Install ceramic sleeve into top vent hole on furnace case.

Retain the original packaging material if re-shipment is foreseen or required.

Installation



Caution

Be sure ambient temperature does not exceed 104°F (40°C). Ambients above this level may result in damage to the controller. Allow at least six inches of space between the furnace and any combustible surface. This permits the heat from the surface case to escape so as not to create a possible fire hazard.

Site Selection

Install furnace on a sturdy surface and allow adequate space for ventilation.

The electrical specifications are located on the specification plate on the back of the furnace. Consult Barnstead International if your electrical service is different than those listed on the specification plate. Prior to connecting your Type F62700 furnace to your electrical supply, be sure the front power switch is in the "OFF" position.



Warning

Do not use in the presence of flammable or combustible chemicals. Fire or explosion may result; this device contains components which may ignite such materials.



Warning

To avoid burns, this furnace must not be touched on the exterior or interior furnace surfaces during use or for a period of time after use.

General Operation

General Operation of Furnace

To avoid electrical shock, this furnace must:

- 1. Use a properly grounded electrical outlet of correct voltage and current handling capacity.
- 2. Disconnect from the power supply prior to maintenance and servicing.
- 3. Have the door switch operating properly.

To avoid personal injury:

- Do not use in the presence of flammable or combustible materials; fire or explosion may result. This device contains components which may ignite such material.
- 2. Refer servicing to qualified personnel.
- 3. Please note the following WARNINGS:

WARNING

This warning is presented for compliance with California Proposition 65 and other regulatory agencies and only applies to the insulation in this product. This product contains refractory ceramic, refractory ceramic fiber or fiberglass insulation, which can produce respirable dust or fibers during disassembly. Dust or fibers can cause irritation and can aggravate pre-existing respiratory diseases. Refractory ceramic and refractory ceramic fibers (after reaching 1000°C) contain crystalline silica, which can cause lung damage (silicosis). The International Agency for Research on Cancer (IARC) has classified refractory ceramic fiber and fiberglass as possibly carcinogenic (Group 2B), and crystalline silica as carcinogenic to humans (Group 1).

The insulating materials can be located in the door, the hearth collar, in the chamber of the product or under the hot plate top. Tests performed by the manufacturer indicate that there is no risk of exposure to dust or respirable fibers resulting from operation of this product under normal conditions. However, there may be a risk of exposure to respirable dust or fibers when repairing or maintaining the insulating materials, or when otherwise disturbing them in a manner which causes release of dust or fibers. By using proper handling procedures and protective equipment you can work safely with these insulating materials and minimize any exposure. Refer to the appropriate Material Safety Data Sheets (MSDS) for information regarding proper handling and recommended protective equipment. For additional MSDS copies, or additional information concerning the handling of refractory ceramic products, please contact the Customer Service Department at Barnstead International at 1-800-553-0039.

Power Switch

Switch power switch to the "I" position. The switch will illuminate when power is on.

Fan

The fan located in the rear of the control section will run continuously as long as power is supplied to the furnace, even when the furnace power switch is OFF. This serves to remove residual heat after the furnace is turned OFF so the heat does not cause damage to sensitive electronic components.

Cycle Indicator

The amber cycle light will illuminate whenever the power is being applied to the elements.

Door Safety Switch

The door safety switch removes power from the heating elements when the door is opened. Open and close door a few times, note that the amber cycle light will go out while the door is open. If this condition is not true, consult the troubleshooting section before proceeding.

Digital Readout

The Digital Readout continuously displays the chamber temperature.



Caution

If the power supply must be disconnected from the furnace at any time, be sure the chamber temperature is 500°C or less before doing so.



Caution

To avoid electrical shock, this furnace must have the door switch operating properly.

Single Setpoint Model w/OTP



Single Setpoint Models

The single setpoint model w/OTP furnace controller is a single setpoint controller which provides a single digital display to indicate the current chamber temperature or setpoint temperature. This temperature controller features sensor break protection, self-tuning capability and over temperature protection (OTP) with an additional OTP relay device.

Basic Operation

When the controller is turned ON it will perform a short self-test and then display the measured value (process value) in the HOME DISPLAY.

Buttons and Indicators

OP1 (Output 1): Illuminates when the logic output is ON.

OP2 (Output 2): Illuminates when the relay output is ON (will go out during an alarm situation).

PAGE button: Allows you to select a new list of parameters.

SCROLL button: Allows you to select a parameter within a list of parameters.

DOWN button: Allows you to decrease a value.

UP button: Allows you to increase a value.



Note

If at any time you want to return to the HOME DISPLAY, simultaneously press PAGE and SCROLL buttons.

To View or Change the Setpoint To view the setpoint, press and release the UP or DOWN

buttons. If you want to change the setpoint, continue pressing until the desired setpoint value is displayed and then release the button. A few seconds after the button is released, the controller will accept the new value and revert to the HOME DISPLAY.

To View the Display Units

From the HOME DISPLAY press the SCROLL button. The display will show the temperature units in °C/F/K and then return to the HOME DISPLAY. (Call Customer Service if you require a different temperature unit.)

To View the % Output Power

From the HOME DISPLAY press the SCROLL button twice. Press and release the UP or DOWN button to view the % output power. This value is a read-only value and cannot be changed.

Controller Parameters

Home display

°C: Temperature units in Celsius. Temperature units can not be changed without entering the configuration. Contact Customer Service if a different temperature unit is required.

OP: % output power demand.

IdHi: Deviation high alarm.

AI List

IdHi: Deviation high alarm.

Atun List

tunE: One-shot autotune enable.

Pid List

Pb: Proportional band (in display units).

ti: Integral time in seconds.

td: Derivative time in seconds.

ACCS List Code: Access code (Code needed to enter or change the other configuration parameters which are not normally accessible.) Not accessible.



Note

The following alarm messages are factory default settings and may vary if you have changed the configuration of your controller:

IDHi = 50°C 2FSH = 1025°C

Alarms

The controller will flash an alarm message in the home display if an alarm condition is detected.

2FSH: Measured value full scale high alarm.

IdHi: Measured value deviation high alarm.

S.br: Sensor break: check that sensor is connected correctly.

L.br: Loop break: check that the heating circuits are working properly.

Ld.F: Heater Circuit fault: indication of either an open or short solid state relay, a blown fuse, missing supply or open circuit heater.

Sensor Break Protection

This controller provides sensor break protection in the event the thermocouple opens. If an open thermocouple condition occurs, the digital display will blink "S.br" and the power to the heating element will be shut OFF (Cycle light will extinguish).

Over-Temperature Protection (OTP)

The OTP will be in effect during any alarm condition when the temperature of the furnace has deviated beyond the limit. The "Deviation High" alarm is the only alarm value which can be changed. To change it, press the SCROLL button until "IdHi" appears on the display. Press the UP or DOWN button to select the OTP value you desire. We recommend a value of 20° above your working temperature to provide protection for your workload.

In addition to over-temperature protection, units containing a single setpoint controller w/OTP feature a mechanical OTP relay device which disconnects power from the elements in an alarm condition.

SINGLE SETPOINT MODEL W/OTP



Note

Start tuning with the process at ambient temperature. This allows the tuner to calculate the low cutback and high cutback values more accurately.



"Stat" and "Sp.rr" in Sp list must be set to OFF or "tunE" will not initiate.

Tuning

This controller incorporates a self-tuning feature which determines the optimum control parameters for the best temperature accuracy with your load and setpoint. Use this feature the first time you use your furnace and each time you change either your setpoint or the type of load you are heating. Barnstead|Thermolyne recommends you use this feature to provide the best temperature accuracy the controller can attain. To use the tuning feature:

- 1. Adjust the setpoint to your desired value.
- 2. Press the PAGE button until display reads, "Atun."
- Press the SCROLL button. Display will read, "tunE."
- 4. Press the UP or DOWN button to select, "on."
- Simultaneously press the PAGE and SCROLL buttons to return to the HOME DISPLAY. The display will alternately flash between "tunE" and the HOME DISPLAY while tuning is in progress.
- 6. The controller will then turn the heating on and off to induce an oscillation. When the measured value reaches the required setpoint the first cycle will end.
- 7. Tuning will be complete after two oscillation cycles and then the tuner will turn itself off.
- 8. Normal control function will resume after the controller calculates tuning parameters.

Single Ramp & Dwell

Note These instructions are used with the Single Setpoint models with OTP only (See models listed on front page).

















Functions

This type of controller has single ramp and dwell programming capabilities. The Ramp and Dwell can be configured to five different modes.

- 1. Mode 1 (Opt. 1) is a Ramp (if needed) to the Setpoint temperature, a Dwell, and then a cool down.
- 2. Mode 2 (Opt. 2) is the same as mode 1, except the controller continues to heat at the Setpoint after the Dwell has completed. (This mode does not cool down.)
- 3. Mode 3 (Opt. 3) is the same as mode 1, except the Dwell time includes the Ramp (if needed).
- 4. Mode 4 (Opt. 4) is the same as mode 2, except the Dwell time includes the Ramp (if needed).
- 5. Mode 5 (Opt. 5) is a Dwell (delay time) before the controller Ramps (if needed) to the Setpoint temperature.

Program Overview

- A program mode can be set by changing the "tm.OP" variable to "Opt. 1, Opt. 2, Opt. 3, Opt. 4, or Opt. 5.
- A Ramp rate may be set by changing the "SPrr" variable to a value. The Ramp rate units are in degrees per minute.
- The Dwell time can be set by changing the "dwEll" variable to the desired value. Dwell time units are in minutes.
- The program Status can be set by changing the "StAt" variable to "run" or "oFF." This variable will start or stop the program.

SINGLE RAMP & DWELL CONTROLLER



Note

The program must be stopped and the controller must be displaying the actual temperature before beginning the Setup.

Program Setup

- Press the PAGE button until the "SP" is displayed. 1.
- 2. Press the SCROLL button once, "SPrr" (Ramp Rate) will be displayed, set the desired Ramp rate with the UP or DOWN buttons, if the ramp to setpoint feature is needed. If the Ramp rate is not needed, then set to "OFF" with the UP or DOWN buttons.
- 3. Press the SCROLL button once, "tm.OP" (Ramp & Dwell mode) will be displayed, select the desired mode with the UP or DOWN buttons. (Opt. 1, Opt. 2, Opt. 3, Opt. 4, Opt. 5)
- Press the SCROLL button once, "dwEll" will be dis-4. played, set the desired Dwell time with the UP or DOWN buttons. (Dwell in minutes.)
- 5. Press the PAGE button until the Actual temperature is displayed.

Running the Program 1. Press the SCROLL button until "StAt" is displayed,

- set to "run" with the UP or DOWN buttons.
- 2. Press the PAGE button to display Actual temperature.

Stopping the Program

Press the SCROLL button until "StAt" is displayed, set to "oFF" with the UP or DOWN buttons.

Clearing the Flashing End

Press the PAGE and SCROLL buttons at the same time.

Verifying a Running Program

Press the SCROLL button until "StAt" is displayed. The display will show "run" if the program is running, or "oFF" if it is not running. Press the PAGE button to display Actual temperature.

8 Segment Programmable Model w/OTP



Note

The controller will return to the HOME DISPLAY if left idle for more than a few seconds.



Note

Once the desired parameter has been selected, depressing either the UP or DOWN button will change the parameter value. In all cases, the value shown on the display is the current working value of that parameter.

Output 1 Display AUTO/MAN Window Button OTHERM EUR 2414 Upper Display Lower Display 2 **RUN/HOLD** Button **UP ARROW** Button PAGE DOWN ARROW SCROLL Button Button Button

The **8 segment programmable** controller consists of a microprocessor based three-mode PID (Proportional, Integral, Derivative), programmable temperature controller with over-temperature protection and appropriate output switching devices to control the furnace. The digital readout continuously displays chamber (upper display) and setpoint (lower display) temperatures unless the SCROLL or PAGE button is depressed. The programmable controller can be used as a single setpoint controller or as a programmable controller. The 8 segment digital model enables eight segments of programming.

Basic Operation

When the controller is turned ON, it will perform a short self-test and then change to the HOME DIS-PLAY. The HOME DISPLAY shows the measured temperature (process value) in the upper display and the desired value (setpoint) in the lower display.

To Change the Setpoint

If you want to change the setpoint, press the UP or DOWN button until the desired setpoint value is displayed in the lower display and then release the button.

To View Display Units

From the HOME DISPLAY press the SCROLL button. The display will briefly show the temperature units in °C/F/K and then return to the HOME DISPLAY. (If you require a different temperature unit call Barnstead International Customer Service.)

To View the % Output Power

From the HOME DISPLAY press the SCROLL button twice. Press the UP or DOWN button to display the value. This value is a read-only value and cannot be changed.

8 Segment Programmable Models with OTP

Buttons and Indicators

OP1 (Output 1): illuminates when the heating output of the temperature controller is on.

AUTO/MAN: (Auto/Manual Mode): when the controller is in the automatic mode the output automatically adjusts to keep the temperature or process value at the setpoint. The "AUTO" light will illuminate. The manual mode has been disabled through factory configuration. Call Customer Service for further information.

RUN/HOLD (Run/Hold button):

- Starts a program when pressed once—RUN light illuminates.
- Holds a program when pressed again—HOLD light illuminates.
- Cancels hold and continues running when pressed again—HOLD light is off and RUN light illuminates.
- Exits a program when the button is held down for two seconds—RUN and HOLD lights are off.
- At the end of a program the RUN light will flash.
- During holdback the HOLD light will flash.

PAGE button: allows you to choose a parameter from a list of parameters.

SCROLL button: allows you to choose a parameter within a list of parameters.

UP button: allows you to increase the value in the lower display.

DOWN button: allows you to decrease the value in the lower display.

Controller Parameters Home Display

°C: measured temperature in Celsius. Temperature units cannot be changed without entering the configuration. Contact Customer Service if a different temperature unit is required.

OP: % output power demand; displayed in lower display (cannot be changed).

C.id: Controller identification number.

IdHi: Deviation High Alarm

tunE: One-shot autotune enable.

run LiSt (Program Run List)

StAt: Displays the program status [OFF, run (running active program), hoLd (program on hold), HbAc (waiting for process to catch up), End (program completed)] in the lower display. The controller will default to "OFF."

FASt: Fast run through program (no/YES). The controller will default to "no."

SEG.d: Flash active segment type in the lower display of the home display (no/YES). The controller will default to "no."

ProG LiSt (Program Edit List)

Hb: Press the UP or DOWN ARROW to select the holdback type [OFF (disables holdback), Lo (deviation low holdback), Hi (deviation high holdback) or bAnd (deviation band holdback)] for the entire program. The controller will default to "OFF."

Hb.U: Press the UP or DOWN ARROW to select the holdback value (in display units). **rmP.U:** Press the UP or DOWN ARROW to toggle between ramp units (SEc, min or Hour). Controller will default to "SEc."

dwL.U: Press the UP or DOWN ARROW to toggle between dwell units (SEc, min or Hour). Controller will default to "SEc."

Cyc.n: Press the UP or DOWN ARROW to set the number of program cycles (1 to 999 or cont). The controller will default to "cont."

SEG.n: Press the UP or DOWN ARROW to select the segment number (1-8 in 8 segment models).

tYPE: Press the UP or DOWN ARROW to select the segment type [End (end of program), rmP.r = ramp rate (ramp to a specified setpoint at a set rate), rmp.t = ramp time (ramp to a specified temperature in a set time), dwEll (to maintain a constant temperature for a set time), StEP (climb instantaneously from current to specified temperature). The controller will default to "End." Other parameters used with tYPE include; tGt target setpoint), Rate (rate of temperature increase) and dur (time to target setpoint or time to dwell)].

End.t: End segment type: dwELL (dwell continuous), rSEt (reset) and S OP (End Segment Output power level.AL LiSt (Alarm List)IdHi: Deviation High Alarm.

Atun LiSt: (Autotune List)

tunE: One-shot autotune enable. **drA:** Adaptive tune enable.

drA.t: Adaptive tune trigger level in display units. Range = 1—9999.

Pid LiSt

G.SP (Gain Setpoint): Is the temperature at which the controller switches from the (SEt 1) PID values to the (SEt 2) PID values.

Pb: Proportional band in display units. (SEt 1)

ti: Integral time in seconds. (SEt 1)

td: Derivative time in seconds. (SEt 1)

Pb2: Proportional band. (SEt 2)

ti2: Integral time in seconds. (SEt 2)

td2: Derivative time in seconds. (SEt 2)

tory default settings and may vary if

you have changed the configuration of

ACCS LiSt (Access List)

Access Code (Code needed to enter or change the other configuration parameters which are not normally accessible.) Call customer service if this configuration is required.



Alarms

The controller will flash an alarm message in the home display if an alarm condition is detected.

IdHi: PV deviation high alarm.

2FSH: PV full scale high alarm.

3FSL: PV full scale low alarm.

LCr: Load current low alarm.

HCr: Load current high alarm.

S.br: Sensor break: check that sensor is connected correctly.

L.br: Loop Break: Check that the heating circuits are working properly.

Ld.F: Heater Circuit Fault: indication of either an open or short solid state relay, a blown fuse, missing supply or open circuit heater.

SSr.F: Solid state relay failure indications in a solid state relay: indicates either an open or short circuit in the SSR.

Htr.F: Heater failure: Indication that there is a fault in the heating circuit: indicates either a blown fuse, missing supply or open circuit heater.

Sensor Break Protection

This controller provides sensor break protection in the event the thermocouple opens. If an open thermocouple condition occurs, the digital display will Blink "S.br" and the power to the heating element will be shut OFF (Cycle light will extinguish).

IDHi = 50°C 2FSH = 1025°C

your controller:

Over-Temperature Protection (OTP)

The OTP will be in effect during any alarm condition when the temperature of the furnace has deviated beyond the limit. The "Deviation High" alarm is the only alarm value which can be changed. To change it, press the SCROLL button until "idHi" appears on the display. Press the UP or DOWN button to select the OTP value you desire. We recommend a value of 20° above your working temperature to provide protection for your workload.

To Operate the Controller as a Single Setpoint Controller

- Switch the circuit breaker to the "ON" position. The setpoint temperature presently set in the controller will appear in the lower display. (The upper display indicates the actual chamber temperature.)
- 2. To change the setpoint, press the UP or DOWN button until the desired setpoint value is displayed; then release the button.
- 3. The furnace will begin to heat if the new setpoint temperature is higher than the present chamber temperature.

Programming the Controller

The controller is capable of varying temperature or process value with time through programming. A program is stored as a series of segments and can be run once, repeated a set number of times or run continuously. To create a customized program using the controller parameters listed under "Controller Parameters" at the beginning of this section, follow the procedures outlined in the proceeding sections of this manual.

Hb: Holdback

Holdback consists of a value and a type. If the measured value lags behind the setpoint by an undesirable amount during a ramp or dwell, the holdback feature can be used to freeze the program at its current state (the HOLD light will flash). The program will resume when the error comes within the holdback value.

OFF: holdback is disabled.

Lo (Deviation Low Holdback): holds the program back when process variable deviates below the setpoint by more than the holdback value.

Hi (Deviation High Holdback): holds the program back when process variable deviates above the setpoint by more than the holdback value.

bAnd (Deviation Band Holdback): combines the features of the high and low deviation holdback in that it holds the program back when the process variable deviates above or below the setpoint by more than the holdback value.

To set the holdback type:

- 1. Press the PAGE button until you reach the program list (ProG LiSt).
- 2. Press the SCROLL button until display reads, "Hb."
- 3. Press the UP or DOWN button to toggle between "bAnd, Hi, Lo and OFF."

Hb U: Holdback Value To set the holdback value:

- 1. Press the PAGE button until you reach the program list (ProG LiSt).
- 2. Press the SCROLL button until display reads, "Hb.U."
- 3. Press the UP or DOWN button to enter a holdback value.



The value set in this parameter is always for the entire program.

rmP.U: Setting Ramp Units

Ramp units are time units which are used in "rmP.r" segments (ramp to a setpoint at degrees per second, minute or hour) and "rmP.t" segments (ramp to setpoint in a specific amount of time). See "Setting the Segment Type" for an explanation on how to set a ramp segment.

- 1. Press the PAGE button until you reach the program list (ProG LiSt).
- 2. Press the SCROLL button until display reads, "rmP.U."
- 3. Press the UP or DOWN button to toggle between seconds, minutes and hours.

dwL.U: Setting Dwell Units

Dwell units are time units which are used in "dwELL" segments (amount of time to remain at a specific temperature). See "Setting the Segment Type" for an explanation on how to set a dwell segment.

- 1. Press the PAGE button until you reach the program list (ProG LiSt).
- 2. Press the SCROLL button until display reads, "dwL.U."
- 3. Press the UP or DOWN button to toggle between seconds, minutes and hours.

CYC.n: Setting the Number of Cycles

Set the number of times a group of segments or programs are to be repeated by following the steps listed below.

- 1. Press the PAGE button until you reach the program list (ProG LiSt).
- 2. Press the SCROLL button until display reads, "CYC.n."
- 3. Press the UP or DOWN button to select the number of cycles you want to run or, press the DOWN button to select "cont." so the program will run continuously.

8 SEGMENT PROGRAMMABLE MODEL W/OTP



Note

The program ramp rate is designed to reduce the heatup rate or cooling rate that the furnace normally exhibits. When not using this feature, the furnace will operate at its maximum heating and cooling capability.



Note

When the program ramp has ended or has been reset, the furnace will continue to maintain setpoint temperature. It will not cool to ambient temperature unless the setpoint is set to ambient temperature by the program or by the operator.

Setting the Segment Type

There are five segment types. Proceed with the following steps according to the type of segment you have selected.

rmP.r (Ramp)

To ramp linearly at a set rate to a specified temperature:

- 1. Press the PAGE button until you reach the program list (ProG LiSt).
- 2. Press the SCROLL button until display reads, "tYPE."
- 3. Press the UP or DOWN button until display reads, "rmP.r."

Steps 4 and 5 are used in the 4 program model only. If you are using an 8 segment program, skip to step 6.

- 4. Press the SCROLL button until display reads "Hb."
- 5. Press the UP or DOWN button to toggle between "bAnd, Hi, Lo and OFF."
- 6. Press the SCROLL button until display reads, "tGt."
- 7. Press the UP or DOWN button to set a target setpoint.
- 8. Press the SCROLL button until display reads, "rAtE."
- 9. Press the UP or DOWN button to select a value in ramp units (seconds, minutes or hours; set in the "rmP.U" parameter).

rmP.t

To ramp to a specified temperature at a set time:

- 1. Press the PAGE button until you reach the program list (ProG LiSt).
- 2. Press the SCROLL button until display reads, "tYPE."
- 3. Press the UP or DOWN button until display reads, "rmP.t."

- 4. Press the SCROLL button until display reads, "tGt."
- 5. Press the UP or DOWN button to set a target setpoint.
- 6. Press the SCROLL button until display reads, "dur."
- 7. Press the UP or DOWN button to select a time in ramp units (seconds, minutes or hours; set in the "rmP.U" parameter.

dwEll

To maintain a constant temperature for a specified time:

- 1. Press the PAGE button until you reach the program list (ProG LiSt).
- 2. Press the SCROLL button until display reads, "tYPE."
- 3. Press the UP or DOWN button until display reads, "dwEll."
- 4. Press the SCROLL button until display reads, "dur."
- 5. Press the UP or DOWN button to select a time in dwell units (seconds, minutes or hours; set in the "dwL.U" parameter).

StEP

To climb instantaneously from the current temperature to a specified temperature.

- 1. Press the PAGE button until you reach the program list (ProG LiSt).
- Press the SCROLL button until display reads, "tYPE."
- 3. Press the UP or DOWN button until the display reads, "StEP."
- 4. Press the SCROLL button until display reads, "tGt."
- 5. Press the UP or DOWN button to set a target setpoint.

End

To end or repeat a program:

- 1. Press the PAGE button until you reach the program list (ProG LiSt).
- 2. Press the SCROLL button until display reads, "tYPE."
- 3. Press the UP or DOWN button until display reads, "End."
- 4. Press the SCROLL button until display reads, "End.t."
- Press the UP or DOWN button to toggle between "dwEll" (an indefinite dwell), "S OP" (End Segment Output Power) and "rSET" (reset).

Running a Program (8 Segment

Programmable Models)

To run a program, press the RUN/HOLD button. (The RUN light will illuminate.)

Holding a Program

To put a running program on hold, press the RUN/HOLD button. (The HOLD light will illuminate.)

Cancelling a Program

To cancel a program, hold the RUN/HOLD button down until the RUN and HOLD lights go off.


Note

Display will flash "tu.ER" if an error occurs during tuning. To clear the error and restart tuning, simultaneously press the PAGE and SCROLL buttons and follow the steps outlined in "Autotuning."



Note

To stop the tuning function, simultaneously press the PAGE and SCROLL buttons.

Tuning

The purpose of tuning your furnace is to match the characteristics of your controller to the characteristics of the process being controlled. Good control is evidenced by: stable, straight-line control of the setpoint temperature with no fluctuations; No overshoot or undershoot of the setpoint temperature; rapid restoration of the setpoint temperature when external disturbances cause deviations from the setpoint.

This controller has automatic tuning features which install optimum tuning parameters to give the best temperature accuracy. No manual loading of tuning parameters is needed. We recommend that you tune the furnace to your specific application to obtain the best results. To provide the best temperature accuracy possible, use these features when you install your furnace and whenever you change your application or procedure.

Tuning Error

The display will flash "tu.ER" if an error occurs during tuning. To clear the error and restart tuning, simultaneously press the PAGE and SCROLL buttons and follow the steps outlined in "Autotuning."

G.SP: Gain Scheduling

Gain scheduling is the automatic transfer of control between two sets of PID values. The controller does this at a presettable process value. Gain scheduling is used for difficult control processes which show large changes in their response time or sensitivity at high or low temperatures, or when heating or cooling.

The G.SP gain schedule setpoint is factory set at 700° C. The G.SP must be adjusted to 200°C from the desired setpoint temperature when tuning.

Setting the Transfer Point

If gain scheduling has been enabled, "G.SP will appear at the top of the PID list. This sets the value at which the transfer will occur. When the process value is below this level, PID1 will be active and when it is above, Pid2 will be active. Set a value between the control regions that show the greatest change to achieve the best point of transfer.

Tuning

The two sets of PID values can be manually set or automatically tuned. To tune automatically you must tune above and below the transfer point G.SP. If the process value is below the transfer point G.SP, the calculated values will automatically be inserted into the (SEt 1) set and if the process value is above G.SP, the calculated values will automatically be inserted into the (SEt 2).



Note

Start tuning with the process at ambient temperature. This allows the tuner to calculate the low cutback and high cutback values more accurately.

Autotuning

The Autotune feature automatically sets up the PID values in the control parameters to suit new process conditions.

To tune your furnace using autotuning:

- 1. Load your furnace with a load similar to your normal load and close the door.
- 2. Set the setpoint temperature.
- 3. Press the PAGE button until the display reads, "Atun LiSt."
- 4. Press the SCROLL button until "tunE OFF" is displayed.
- 5. Press the UP or DOWN button to select "on."
- Simultaneously press the PAGE and SCROLL buttons to return to the HOME DISPLAY. The display will flash "tunE" while tuning is in progress.

Adaptive Tuning

Adaptive tuning continuously evaluates tuning parameters. Adaptive tuning automatically installs new values if better accuracy is possible. Adaptive tuning should be used when the characteristics of a process change due to load or setpoint changes or, in a process that can not handle the oscillation caused by a one-shot tune.

To tune your furnace using adaptive tuning:

- 1. Load your furnace with a load characteristic of those you intend to heat in it.
- 2. Press the PAGE button until display reads, "Atun LiSt."
- 3. Press the SCROLL button until "drA OFF" is displayed.
- 4. Press the UP or DOWN button to select "on."
- 5. Press the SCROLL button until "drA.t" is displayed.
- 6. Press the UP or DOWN button until the desired trigger value is achieved.

Furnace Loading



Caution

Do not overload your furnace chamber. If the load is to be heated uniformly it should not occupy more than the center two-thirds of the furnace chamber. Failure to observe this caution could result in damage to furnace components.

- 1. For best results use only the center 2/3 of the furnace chamber.
- 2. If you are heating a number of small parts, spread them throughout the center two-thirds of the furnace chamber.
- 3. Keep objects away from thermocouple.
- 4. Use insulated tongs and mittens when loading and unloading furnace.
- 5. Always wear safety glasses.
- 6. Use the hearth plates supplied to protect bottom of chamber. Part # PH421X1.
- 7. Do not exceed a load of 25 lbs. in the furnace chamber.

Preventative Maintenance

Please note the following WARNINGS:

WARNING

This warning is presented for compliance with California Proposition 65 and other regulatory agencies and only applies to the insulation in this product. This product contains refractory ceramic, refractory ceramic fiber or fiberglass insulation, which can produce respirable dust or fibers during disassembly. Dust or fibers can cause irritation and can aggravate pre-existing respiratory diseases. Refractory ceramic and refractory ceramic fibers (after reaching 1000°C) contain crystalline silica, which can cause lung damage (silicosis). The International Agency for Research on Cancer (IARC) has classified refractory ceramic fiber and fiberglass as possibly carcinogenic (Group 2B), and crystalline silica as carcinogenic to humans (Group 1).

The insulating materials can be located in the door, the hearth collar, in the chamber of the product or under the hot plate top. Tests performed by the manufacturer indicate that there is no risk of exposure to dust or respirable fibers resulting from operation of this product under normal conditions. However, there may be a risk of exposure to respirable dust or fibers when repairing or maintaining the insulating materials, or when otherwise disturbing them in a manner which causes release of dust or fibers. By using proper handling procedures and protective equipment you can work safely with these insulating materials and minimize any exposure. Refer to the appropriate Material Safety Data Sheets (MSDS) for information regarding proper handling and recommended protective equipment. For additional MSDS copies, or additional information concerning the handling of refractory ceramic products, please contact the Customer Service Department at Barnstead International at 1-800-553-0039.

This unit is equipped with a venting system on the top of the furnace. This is for the removal of fumes from the chamber of the unit. Contamination is a major cause of element failure, therefore, when possible remove the fume forming material before heating. (e.g., cleaning cutting oil from tool steel.)

Housekeeping is vital to your electric furnace - KEEP IT CLEAN. Run your furnace up to 871°C empty occasionally to burn off the contamination that may exist on the insulation and elements. Maintain 871°C for at least 4 hrs.



Warning

Disconnect from the power supply prior to maintenance and servicing. Refer servicing to qualified personnel. to insure complete ashing of foreign materials.

Element life is reduced somewhat by repeated heating and cooling. If the furnace is to be used again within a few hours, it is best to keep it at the operating temperature or at a reduced level such as 260°C.

General Cleaning Instructions Wipe exterior surfaces with lightly dampened cloth con-

taining mild soap solution.

Troubleshooting

The Troubleshooting section is intended to aid in defining and correcting possible service problems. When using the chart, select the problem category that resembles the malfunction; then proceed to the possible causes category and take necessary corrective action.

PROBLEM	POSSIBLE CAUSES	CORRECTIVE ACTION
The power light does not illuminate.	The furnace is not connected to power supply.	Check furnace connection to power source.
	ON and OFF power switch defective.	Replace power switch.
Fan does not operate.	The furnace is not connected to the power supply.	Check furnace connection to power source.
	Blown fuses.	Replace fuses.
The furnace does not heat.	No power.	Check power source and fuses or breakers.
	Thermocouple is open or thermocouple leads reversed.	Replace thermocouple or check thermocouple connections.
	Controller malfunction.	Verify and correct all parameters and configuration values. If malfunction persists, replace control.
	Element burned out.	Replace muffle (element).
	Solid state relay defective.	Replace solid state relay.
	Door switch malfunction.	Re-align or replace door switch.
Slow heatup.	Low line voltage.	Install line of sufficient size and proper voltage. (Isolate furnace from other electrical loads.)
	Heavy load in chamber.	Lighten load in chamber to allow heat to circulate.
	Wired improperly.	Check wiring diagram for correct wiring of your furnace.
	One side of element is burned out on 120V or 100V unit.	Replace muffle (element).

TROUBLESHOOTING

PROBLEM	POSSIBLE CAUSES	CORRECTIVE ACTION
Furnace doesn't stop heating when the door is opened.	Door switch is malfunctioning.	Re-align or replace door safety switch.
	Mechanized relay is malfunctioning.	Replace relay.
Repeated element burnout.	Heating harmful materials.	Clean up spills in and on chamber. Ventilate chamber by leaving top vent slightly open when heating known harmful reagents.
	Wrong element.	Install proper element.
	Oxidized thermocouple.	Replace thermocouple.
	Contamination present from previous burnout.	Clean and/or replace insulation material.
	Wired improperly.	Check wiring diagram for correct wiring of your furnace.
Inaccurate temperature readout.	Oxidized or contaminated thermocouple.	Replace thermocouple.
	Poor thermocouple connection.	Tighten connections.
	Solid state relay malfunction.	Replace solid state relay.
	Improper loading procedures.	Use proper loading procedures.
	Poor ventilation of base or cooling fan failed.	Clear area around furnace base or replace cooling fan.
	Control out of calibration.	Contact Barnstead International.
	Thermocouple connections reversed.	Reconnect thermocouple correctly.
	P.I.D. values invalid.	Re-tune control.
	Control malfunction.	Verify and correct all parameter and configuration values. If malfunction persists, replace control.

Maintenance and Servicing

Please note the following WARNINGS:

WARNING

This warning is presented for compliance with California Proposition 65 and other regulatory agencies and only applies to the insulation in this product. This product contains refractory ceramic, refractory ceramic fiber or fiberglass insulation, which can produce respirable dust or fibers during disassembly. Dust or fibers can cause irritation and can aggravate pre-existing respiratory diseases. Refractory ceramic and refractory ceramic fibers (after reaching 1000°C) contain crystalline silica, which can cause lung damage (silicosis). The International Agency for Research on Cancer (IARC) has classified refractory ceramic fiber and fiberglass as possibly carcinogenic (Group 2B), and crystalline silica as carcinogenic to humans (Group 1).

The insulating materials can be located in the door, the hearth collar, in the chamber of the product or under the hot plate top. Tests performed by the manufacturer indicate that there is no risk of exposure to dust or respirable fibers resulting from operation of this product under normal conditions. However, there may be a risk of exposure to respirable dust or fibers when repairing or maintaining the insulating materials, or when otherwise disturbing them in a manner which causes release of dust or fibers. By using proper handling procedures and protective equipment you can work safely with these insulating materials and minimize any exposure. Refer to the appropriate Material Safety Data Sheets (MSDS) for information regarding proper handling and recommended protective equipment. For additional MSDS copies, or additional information concerning the handling of refractory ceramic products, please contact the Customer Service Department at Barnstead International at 1-800-553-0039.

MAINTENANCE AND SERVICING



Warning

Disconnect from the power supply prior to maintenance and servicing. Refer servicing to qualified personnel. Replace fuses with the same type and rating.



Note

Perform only maintenance described in this manual. Contact an authorized dealer or our factory for parts and assistance.



Note

Make sure yellow thermocouple lead wire is connected to terminal V+ on back of the controller. Make sure red thermocouple lead wire is connected to terminal V- on back of the controller. Make sure the exposed section of the T/C lead wires are not contacting each other.

Fuse Replacement

- a. Disconnect from power supply.
- b. Set furnace on its door.
- c. Remove (one piece) top, back and bottom cover.
- d. Replace fuses.

Thermocouple Replacement (Type K)

- a. Disconnect from power supply.
- b. Set furnace on its door.
- c. Remove (one piece) top, back and bottom cover.
- d. Remove retaining clip from the thermocouple and pull the thermocouple straight out from the chamber.
- e. Disconnect thermocouple lead ends from back of the controller and remove the thermocouple.
- f. Reinstall thermocouple by reversing steps A through E.

Muffle (Heating Element) Replacement

- a. Disconnect from power supply.
- b. Set furnace on its door.
- c. Remove (one piece) top, back and bottom cover.
- d. Disconnect element lead wires from relays. (Note placement and connection of.)
- e. Remove clip from thermocouple and pull thermocouple out of muffle.
- f. Remove metal retaining band from defective muffle.



Note

Make sure the black insulation sleeving covers the exposed portion of the element lead wires.



Note

Make sure the exposed section of the T/C lead wires are not contacting each other.

- Loosen top adjustment bracket and remove deg. fective muffle.
- h. Insert new muffle with vent hole facing the top of unit.
- Readjust top bracket to fit snugly up against i. muffle. Make sure muffle is laying flush against front case section.
- Reinstall metal retaining band and tighten until j. muffle seats firmly into case section.
- k. Reconnect element lead wires.

On the 120V or 100V muffle there are three element lead wires. Connect the front and the back element lead wires to the mechanical relay. Connect the middle element lead wire to the solid state relay.

On the 230V and 240V muffles there are two element lead wires. Connect the front element lead wire to the mechanical relay. Connect the back element lead wire to the solid state relay.

- Ι. Reinstall the thermocouple and the retaining clip.
- Reinstall the cover section. m.
- Set the furnace upright and test the operation of n. the furnace.

Wiring Diagrams

Single Setpoint w/OTP Control



8 Segment Programmable Control



Replacement Parts

Listed below are the common replacement parts for all models of the F62700 furnace.

PART	PART NO.
Temperature Control (Single Set Point Models)	CN71X76
Temperature Control (8 Segment Programmable Models)	CN71X77
Muffle Heating Element 100V	EL1075X3A
Muffle Heating Element 120V	EL1075X1A
Muffle Heating Element 220-240V	EL1075X2A
Solid State Relay	RYX34
Mechanical Relay 120V, 100V	RYX56
Mechanical Relay 220-240V	RYX57
On/Off Switch 120V	SWX143
On/Off Switch 220-240V	SWX144
Fan 120V, 100V	FAX29
Fan 220-240V	FAX7
Door Switch	SW1172X1
Thermocouple	TC517X1A
Door Insulation	JC517X1
Hearth Plate (2 required)	PH421X1
Pilot light, 100V & 120V	PLX76
Pilot light, 220-240V	PLX82
Door Assembly, with Insulation	DR1075X1A
Fuses, Type Non-Time lag, 250 Volt, 10 Amp (F62730 Models)	FZX30
Fuses, Type Non-Time lag, 250 Volt, 20 Amp (F62734, F62735 Models)	FZX29



Warning

Replace fuses with the same type and rating.

Ordering Procedures

Please refer to the Specification Plate for the complete model number, serial number, and series number when requesting service, replacement parts or in any correspondence concerning this unit.

All parts listed herein may be ordered from the **Barnstead International** dealer from whom you purchased this unit or can be obtained promptly from the factory. When service or replacement parts are needed we ask that you check first with your dealer. If the dealer cannot handle your request, then contact our Customer Service Department at 563-556-2241 or 800-553-0039.

Prior to returning any materials to **Barnstead International**, please contact our Customer Service Department for a "Return Materials Authorization" number (RMA). Material returned without an RMA number will be refused. Minimum invoice: \$25.

Material Safety Data Sheet

Thermal Ceramics Material Safety Data Sheet

Date Revised: 7/2/91

PRODUCT IDENTIFICATION

Trade Name(s):	CERAFIBER					
Generic Name:	REFRACTORY	CERAM	IIC FIBER INSU	JLATION	CAS #:	65997-17-3
Chemical Name:	ALUMINA SILIC	CA			Formula:	MIXTURE
Manufacturer:	Thermal Ceram	ics			Telephone:	(404) 796-4200
Address:	P.O. BOX 923,	2102 Ol	d Savannah Ro	ad		
City:	Augusta	State:	Georgia Zip:	30903		

PRODUCT INGREDIENTS

Ingredient Name	Cas Number	%	PEL and TLV (except as noted)
REFRACTORY CERAMIC FIBER	65997-17-3	100	1 FIBER/CC EXPOSURE GUIDELINE 5mg/M3 - NUISANCE RESPIRABLE - OSHA 10mg/M3 - NUISANCE TOTAL - ACGIH
CRYSTALLINE SILICA (CRISTOBALITE) WILL FORM "AFTER SERVICE" AT TEMPERATURES >1000°C.	14464-46-1	>20	0.05 mg/M3 - OSHA Respirable Dust

PHYSICAL DATA

Appearance and Odor:	WHITE FIBER-NO ODOR.		
Boiling Point:	NA	Evaporation Rate (NA = 1):	NA
Vapor Pressure:	NA	Specific Gravity (water = 1):	2.6
Water Solubility (%):	NIL	Melting Point: >3000°F	
Vapor Density (Air= I):	NA	% Volatile by Volume: 0	

FIRE AND EXPLOSION DATA

Flash Point (Method):	NONFLAMMABLE	NFPA Flammable/Combustible Liquid				
Classification:	NA	Flammable Limits:	LEL:	NA %	UEL:	NA %
Auto-Ignition Temperature:	NA					

HEALTH HAZARDS

Summary/Risks

- Summary: EXPOSURE TO DUST FROM THIS PRODUCT SHOULD BE MINIMIZED. ANIMAL INHALATION AND ARTIFICIAL IMPLANTATION STUDIES HAVE REPORTED THE DEVELOPMENT OF TUMORS. BASED ON PRELIMINARY RESULTS, A NOTICE OF SUBSTANTIAL RISK HAS BEEN FILED WITH THE EPA ACCORDING TO SECTION 8(e) OF THE TOXIC SUBSTANCES CONTROL ACT. BASED ON ANIMAL STUDIES, IARC HAS CLASSIFIED RCF AS POSSIBLY CARCINOGENIC FOR HUMANS (2B). DATA FROM HUMAN EPIDEMIOLOGICAL STUDIES IS INSUFFICIENT. THIS SUBSTANCE OR MIXTURE HAS NOT BEEN CLASSIFIED A CARCINOGEN BY NTP OR OSHA.
- Medical conditions which may be aggravated: AS WITH ANY DUST, PRE-EXISTING UPPER RESPIRATORY AND LUNG DISEASES MAY BE AGGRAVATED.

Target Organ(s): LUNGS, SKIN AND EYES.

Acute Health Effects: PRODUCT IS A MECHANICAL IRRITANT TO SKIN, EYES AND UPPER **RESPIRATORY SYSTEM.**

Chronic Health Effects: EXCESSIVE EXPOSURE TO RCF DUSTS AND AFTER SERVICE FIBERS MAY CAUSE LUNG DAMAGE (FIBROSIS). IARC STATES THERE IS SUFFICIENT EVIDENCE IN ANIMALS AND LIMITED EVIDENCE IN HUMANS TO CLASSIFY CRYSTALLINE SILICA AS A PROBABLE CARCINOGEN (2A) AND RCF AS A POSSIBLE CARCINOGEN (2B).

Primary Entry Route(s): INHALATION, SKIN AND EYE CONTACT.

Signs/Symptoms of Overexposure

Inhalation: IRRITATION OR SORENESS IN THROAT & NOSE. IN EXTREME EXPOSURES SOME CONGESTION MAY OCCUR.

Skin Contact: TEMPORARY IRRITATION OR RASH.

Skin Absorption: NA

Ingestion: NOT HAZARDOUS WHEN INGESTED. MAY CAUSE TEMPORARY IRRITATION TO GI TRACT. Eyes: TEMPORARY IRRITATION OR INFLAMMATION.

First Aid/Emergency Procedures

Inhalation: REMOVE TO FRESH AIR. DRINK WATER TO CLEAR THROAT AND BLOW NOSE TO EVACUATE FIBERS.

Skin Contact: WASH AFFECTED AREAS GENTLY WITH SOAP AND WARM WATER.

Skin Absorption: NA

Indestion: NA

Eyes: FLUSH EYES WITH COPIOUS QUANTITIES OF WATER. IF IRRITATION PERSISTS CONSULT A PHYSICIAN.

REACTIVITY DATA

MATERIAL IS STABLE. HAZARDOUS POLYMERIZATION CANNOT OCCUR. Chemical Incompatibilities: HYDROFLUORIC ACID Conditions to Avoid: NONE IN DESIGNED USE. Hazardous Decomposition Products: NONE

SPILL OR LEAK PROCEDURES

Procedures for Spill/Leak: VACUUM CLEAN DUST WITH EQUIPMENT FITTED WITH HEPA FILTER. IF SWEEPING IS NECESSARY USE A DUST SUPPRESSANT.

Waste Management: WASTES ARE NOT HAZARDOUS AS DEFINED BY RCRA (40 CFR PART 261). COMPLY WITH FEDERAL, STATE & LOCAL REGULATIONS. METHOD OF DISPOSAL - LANDFILL, RQ - N/A.

SPECIAL PROTECTION INFORMATION

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- Respirator: <1 F/CC, USE 3M 9900; <10 F/CC, USE MSA COMFO II WITH H FILTER; <50 F/CC, USE MSA ULTRA-TWIN H FILTER; OR EQUIVALENTS. SEE SECTION IX-OTHER.
- Ventilation: USE SUFFICIENT NATURAL OR MECHANICAL VENTILATION TO KEEP DUST LEVEL TO BELOW PEL/TLV/WEG (WORKPLACE EXPOSURE GUIDELINE) USE DUST COLLECTION WHEN TEARING OUT.

Other: WEAR LOOSE FITTING, LONG SLEEVED CLOTHING. WASH EXPOSED AREAS WITH SOAP & WARM WATER AFTER HANDLING. WASH WORK CLOTHES SEPARATELY FROM OTHER CLOTHING; RINSE WASHER THOROUGHLY.

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Special Handling/Storage: KEEP MATERIAL DRY.

Special Workplace Engineering Controls: ADEQUATE VENTILATION TO KEEP DUST LEVEL TO BELOW PEL/TLV/WEG (WORKPLACE EXPOSURE GUIDELINE).

Other: ADDITIONAL INFORMATION ON THE HEALTH AND SAFETY ASPECTS OF REFRACTORY CERAMIC FIBERS IS AVAILABLE.

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Section 7 – Kohl and Nishiizumi, 1992

Scientific Comment

Chemical isolation of quartz for measurement of *in-situ*-produced cosmogenic nuclides

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Abstract—An isolation method relying totally on chemical steps was developed to separate large quantities (10-200 g) of clean mono-minerallic quartz samples from a variety of terrestrial rocks and soils for the purpose of measuring ${}^{10}\text{Be}(t_{1/2} = 1.5 \text{ Myr})$ and ${}^{26}\text{Al}(t_{1/2} = 0.705 \text{ Myr})$ produced by cosmic rays *in situ* in the quartz phase. The procedure consists of grinding the sample, heating it in HCl, and treating it with a series of leaches using a dilute HF/HNO₃ mixture in a heated ultrasonic tank. The purified quartz was also used for the measurements of *in-situ*—cosmic-ray-produced ${}^{21}\text{Ne}$ and ${}^{14}\text{C}(t_{1/2} = 5730 \text{ yr})$. The method is applicable to any problem requiring purified quartz on a large scale.

INTRODUCTION

MEASUREMENT OF COSMOGENIC nuclides produced in situ in terrestrial samples shows great potential as a tool for quantifying continental erosion rates, determining exposure ages of rocks, dating various geologic events, and elucidating past climates (LAL and ARNOLD, 1985). Quartz, which is geologically abundant and relatively easy to separate from other minerals, is ideal for these studies. Its advantages include a tight crystal structure which minimizes possible contamination by meteoric ¹⁰Be, produced in the atmosphere, and a simple target chemistry, Si and O, which allows straightforward theoretical calculations of ¹⁰Be and ²⁶Al production rates. The primary reason for selecting quartz for this work is its low Al content which allows measurement of ²⁶Al after exposures as short as 10³ years. For ²⁶Al to be accurately determined by accelerator mass spectrometry (AMS), the ²⁶Al/²⁷Al ratio must be greater than 10⁻¹⁴. The purification technique described here produces quartz with a stable Al (²⁷AI) content sufficiently low to allow ²⁶Al measurements to be made, eliminates any possible contamination by meteoric ¹⁰Be, and effectively allows processing of the large quantities (10-200 g) of quartz needed.

The feasibility of quantitatively measuring ¹⁰Be and ²⁶Al in terrestrial quartz has been demonstrated (NISHIIZUMI et al., 1986), and the method has been calibrated both by measurements of ¹⁰Be and ²⁶Al in rocks of known exposure (NI-SHIIZUMI et al., 1989) and by laboratory experiments using bombardment by high-energy neutrons and μ^- (Reedy et al., unpubl. data). The AMS measurements require 10-200 g of purified quartz, depending on exposure age and altitude and on Al content. Previous separation methods produce only a few hundred mg of quartz (SYERS et al., 1968). These schemes have relied on heavy liquid density separations, NaHSO4 fusions, and NaOH and H2SiF6 leaches (HENDERSON et al., 1972; SRIDHAR et al., 1975; SYERS et al., 1968). Heavy liquids are expensive although they can be recycled. Unfortunately, however, many of them do attack some mineral grains, dissolving a variety of elements and creating unacceptable potential levels of cross contamination. Separation of 100 g or so of quartz from a large amount of other minerals would be unwieldy. Two density separations would be required to remove minerals with higher and lower densities than quartz and many feldspars (a major high-Al mineral phase in many rock types) have densities similar to quartz, 2.65 g/cm³. The plagioclase (Na, Ca) feldspars have densities in the range 2.62-2.76 g/cm³, and the K feldspars have densities of 2.56 g/cm³. In addition, density separations cannot eliminate meteoric ¹⁰Be from the quartz; such purification requires an etching step of some sort. The NaHSO4 fusion does not scale to the large sample sizes required for ¹⁰Be-²⁶Al work; for example, the 300 g of powdered rock routinely processed would require 18 kg of NaHSO4 based on the previous work of SYERS et al. (1968) and SRIDHAR et al. (1975). H₂SiF₆ is not as effective as HF in dissolving extraneous minerals; in addition, the commercially available reagent has a high Al content (50 ppm). Although the procedures described here probably require as much time as other methods to complete preparation of a quartz sample, the previous methods with their large number of steps require continuous attention, unlike this simplified procedure. Since studies of continent-wide processes necessitate measurement of large numbers of samples, it is important to have a separation procedure which is simple, routine, and flexible and which produces a high yield of clean quartz from a variety of different rock and soil types. In addition, the method is applicable to any problem requiring purified quartz on a large scale.

EXPERIMENTAL

The separation procedure developed here to isolate quartz from other minerals depends on its low reactivity with various acids, except in the presence of fluoride. The techniques are completely chemical and are adaptable to large sample sizes.

The initial assay for quartz purity was visual observation under a microscope. The routine quantitative test was Al analysis performed by atomic absorption spectrophotometry on a completely dissolved aliquot of the separated material. As an additional check for the presence of extraneous mineral phases, X-ray diffraction was run on a few selected samples. Electron microprobe analysis could not be used because the detection limit for this technique is too high, $\sim 0.1\%$ Al. Yields of quartz were monitored by processing pure rock crystal quartz, ground to different grain sizes, through the various chemical procedures.

As a first step, the rocks or soils were crushed, as necessary, using a jaw crusher, and were ground using a pulverizer to a particle size designed to produce mostly mono-minerallic grains of as large a size as possible. If the sample had large crystals, these grains were 1-2mm in diameter. The majority of samples were ground to $\leq 700 \ \mu m$. The first chemical treatment was designed to eliminate easily dissolved minerals without losing any quartz. It consists of heating the ground sample for 12–24 h in 1:1 (by volume) HCl (\sim 1 g sample per 10 mL) with \sim 0.03% of H₂O₂. This procedure mainly dissolves carbonates and iron oxides. The residue was washed several times with deionized H₂O, and fines were discarded with the rinse water.

A variety of different acid mixtures and agitation sequences was tried to determine the most effective method of eliminating unwanted silicate minerals and to maximize the purity and yield of the final quartz. Qualitative tests (microscope examination of product) using dilute mixtures of HF/H₂SO₄, HF/HNO₃ and HF/HCl as the leachant established HF/HNO₃ as the most promising. Preliminary tests involved boiling relatively small amounts of sample in the acid mixture under study for 30 min to 2 h. The rock used in these tests was a pegmatite collected from the desert at Anza Borrego, California, ground to grain sizes between 70 and 700 μ m. Using this method, 1% HF/10% HNO₃ was found to give the same purification (Al content) as and a higher yield than 1% HF/35% HNO₃ or than 3% HF/10% HNO₃.

To scale the procedure to sample sizes of several hundred grams acid treatments were devised in which the polypropylene bottles or beakers containing the sample-acid mixture were agitated in a water bath heated to ~95°C. The agitation was accomplished by placing the beakers in a heated ultrasonic tank which was run continuously for the duration of the leach or by placing vented bottles in a shaker assembly which vigorously shook them 1 minute out of 10. The shaker had to move the bottles a distance of \sim 5 cm at a rate of one hundred times a minute to give separations which equalled those of the ultrasonic bath. We currently use the ultrasonic system exclusively because it gives much less mechanical trouble and we are able to run the equipment every day without breakdowns. Table 1 lists a number of different leaches run on a variety of rock and samples with various concentrations of HF and HNO3; ratios of sample to HF and HNO3; and, in some cases, the addition of NH4F or HClO4. A similar pegmatite to the earlier work was used as a reference sample for the majority of these investigations. Yields are defined as weight of material after the leach step divided by weight of material at the start of the leach.

There is a dramatic reduction in the Al content after one 9-h leach (Fig. 1) for samples run at ratios of 7.5 g sample/L of 1% HF acid mixture or less. This represents a slight excess of HF over the stochiometric amount needed to dissolve all the sample if it were completely SiO_2 . If there is more sample than HF, the Al-containing

TABLE 1. Leaching Tests

Sample Grai	n Size W	eight	Тіте Вил	Lcac HE	hant HNO2	Yield ¹)	AI
(LL)	m) (m	(g/l)	(hours)	(%)	(%)	(%)	(mgg)
Pegmatite (A	nza Borre	go)					
Č <15	00	6	12	1	10	23	98
<15	00	7.5	12	1	10	24 not	measured
<15	00	9	12	1	10	25	4000
<15	60	9	12	2	10	21	90
<15	60	18	12	I	10	58	22000
<15	60	18	12	2	10	46	15000
<15	60	18	12	5	10	20	610
<15	60	18	12	10	10	20	25000
<15	60	6	12	02)	0	82	>>1000
<15	600	6	12	13)	10	20	110
200	-600	7.5	9	1	0.5	25	90
200	-600	7.5	9	1	0.1	24	120
200	-600	7.5	9	1	0	25	110
200	-600	7.5	9	14)	1	24	61
600	-1500	15	9	1	1	30	1540
600	-1500	7.5	9	I	1	29	140
600	-1500	5	9	I	1	27	97
Sandstone (W	Volfe Cree	k Crat	cr)				
400	⊢1200	7.5	· 9	1	10	66	310
400	F1200	7.5	9	1	5	68	250
400	-1200	7.5	9	1	1	68	300
Sand (Great S	Sandy De:	sert)					
400	1-700 ⁱ	7.5	9	L	10	91	51
400	-700	7.5	9	I	5	91	53
400	⊢700	7.5	9	1	2.5	92	30
400	F100	7.5	9	1	1	91	55

yield= material after processing/ material before processing

2) includes 2.6% NH₄F

3) includes 2.4% NH4F

4) includes 3.5% HClO₄



FIG. 1. (a) Yields of quartz as a function of time run for the 1% HF-1% HNO₃ leaches. All samples are run at 7.5 g sample/L of leachant unless noted. Zero hours run corresponds to the sample after the HCl step. Yields are defined as undissolved material at each point in time divided by the amount at the start of the run. It is interesting to note that there is little further attack on the pure quartz once extraneous minerals are dissolved, even after six additional hours of leaching. (b) The purification of quartz, measured by the Al content, as a function of time for the 1% HF-1% HNO₃ leaches. All samples are run at 7.5 g sample/L of leachant unless noted. These are the same sample runs as plotted in Fig. 1a.

minerals are not nearly as effectively removed. For the pegmatite (200-600 μ m), the sand, and the sandstone samples, the yields and Al contents were substantially the same for HNO₃ contents from 10 to 1%. Although yields and Al content were good for 0.1% and 0% HNO3 for the pegmatite, there was a large amount of suspended white matter in the leachant. There is often a fine white powder found sticking to the sides of the plastic beakers at the ends of the runs. This material may coat the grains of quartz as well and possibly interfere with further.HF attack. An attempt to keep this material in solution involved adding a small amount (3.5%) of HClO4 to the leachant; however, this did not improve the separation. Addition of NH₄F to the leachant, likewise, had little effect on the final purification. The final established procedure is a 9-h leach in 1% HF/1% HNO₃ at a ratio of 7.5 g sample/L of leachant. After leaching, the acid was decanted, then the samples were rinsed several times with deionized H₂O and dried. Fines always contained higher concentrations of Al than the coarser material and were discarded with the rinse water.

Figures 1a,b show the effectiveness of the leaching as a function of time run for several different types of samples at the ratio of 7.5 g/L leachant. In the case of the reference pegmatite (grain size 0.6-1.5 mm), two different ratios of sample to acid mixture, 7.5 g/L and 15 g/L, are shown. These tests were run in an ultrasonic tank which

takes about 1 h to heat up to ~95°C. The 1% HF/1% HNO3 acid mixture was used for all tests. Yields (Fig. 1a) are calculated as weight of undissolved mineral at a specific time divided by the weight of sample at the start of the run. The Al concentrations (Fig. 1b) are measured from independent aliquots of each ground sample run separately for the various lengths of time. Therefore, it is not surprising that in some cases there is an apparent increase of Al with time run. Although the yields (Fig. 1a) remain nearly constant for most of the samples after the first 3-4 h of processing, indicating that there is no major attack on the remaining quartz, the Al concentrations (Fig. 1b) in the samples do, in some cases, decrease continuously during the subsequent 4-5 h of processing. The greater effectiveness of the smaller ratio of sample to acid solution, 7.5 g/L vs. 15 g/L, is clear from these figures. Although not shown on the figure, a test on the same pegmatite using 1% HF/10% HNO3 gives not only the same final yield and Al content as the 1% HF/1% HNO3 acid leachant but the same pattern over time run as well.

Yields from each step in the purification process are shown in Fig. 2a for a variety of sample types. The 1% HF/1% HNO₃ acid mixture was used for all tests, at the ratio 7.5 g sample/L leachant. In this figure, yield is cumulative, defined as remaining mineral compared to bulk sample. The flatness of the curves for the later steps means that only a small amount of mineral (quartz) is being dissolved by the continued processing. The final yield of purified quartz of course



FIG. 2. (a) Yields of purified quartz for successive processing steps for a variety of sample types. All samples are run at a ratio of 7.5 g sample/L of leachant. The leachant used is 1% HF-1% HNO₃ in all cases. Yields here are cumulative, calculated as weight of undissolved mineral (quartz) after each given step divided by the weight at the start of the HCl step. (b) Changes in the Al content of the purified quartz as a function of the processing steps. All samples are run at a ratio of 7.5 g sample/L of leachant, which is 1% HF-1% HNO₃ in all cases. The samples and runs are the same as in Fig. 2a.

TABLE 2. Quartz Yield* Tests in Percent

1:1 HCI	1%HF/10%HNO3	1%HF/1%HNO3
leach	9 Hour Ultrasonic	9 Hour Ultrasonic
99.8	95.4	94.2
99.9	91.7	92.5
99.9	89.6	90.5
99.1	84.7	82.3
98.6	72.7	73,5
97.1	.49.7	44.7
	1:1 HCl leach 99.8 99.9 99.9 99.1 98.6 97.1	1:1 HCI 1%HF/10%HNO3 <u>leach</u> 9 Hour Ultrasonic 99.8 95.4 99.9 91.7 99.9 89.6 99.1 84.7 98.6 72.7 97.1 49.7

*All yields calculated separately; yield = material after each step/ material before that step

depends on the amount of quartz present in the rock. To investigate the effect of the procedure on pure quartz, yield tests were run with rock crystal quartz ground to different particle sizes. The results are shown in Table 2 for the HCl step and for two different acid leachant mixtures. The loss during the HCl step is probably due to loss of fine powder during transferring. Since the ratio of sample to acid was the samples are attacked before the quartz, these yields represent minima. The total amount of quartz dissolved depends on the grain size as shown, with finer particles being dissolved more readily. Some attempts were made to adapt this purification scheme to very fine (≤ 30 μ m) quartz particles, such as would be found in pelagic sediments, with only moderate success.

Al content vs. step for the same test runs as plotted in Fig. 2a is presented in Fig. 2b. After two to four ultrasonic leaches (the ratio of acid to sample was reduced by a factor of 2 to 3 for the second to fourth leaches), the Al reaches a constant level which represents the actual Al concentration in the quartz grains. Al content in quartz varies from a few to several 1000 ppm, depending on the origin of the quartz (LEHMANN and BAMBAUER, 1973). X-ray diffraction of quartz (3rd ultra) from a selected sandstone (not plotted), Al content 350 ppm, gives an ideal quartz spectrum with no extraneous lines. To check the Al concentration in the purified quartz from the granite, an aliquot of the sample from the second ultrasonic treatment (Al content of 60 ppm) was further ground and leached in an additional ultrasonic step. The Al concentrations in different particle sizes, 70-600 μ m and <70 μ m are very similar to that found for the original unground sample (42, 45, and 52 ppm respectively, after the third leach), indicating that the Al is homogeneously distributed throughout the quartz grains.

In addition to Al, several other elements were measured in the purified quartz. Over one hundred different rock, sand, and soil samples have been treated with the procedures described here. Concentration ranges for the purified quartz obtained are as follows: 0.1-50 ppm for Mg, 0.5-50 ppm for K, 0.5-30 ppm for Ca, and 0.5-200 ppm for Fe compared to 15-400 ppm for Al, with a very few samples exceeding these values. There is some correlation between increased Al and increases in the other elements. The amount of mineral from the final quartz separate which could not be dissolved in 49% HF was usually less than 0.01% and always less than 0.1%. This was sufficient purification for the *in situ* ¹⁰Be-²⁶Al determinations for which this purification scheme was designed. However, an appropriate density separation following further grinding of the purified quartz might reduce the amount of undissolvable mineral if it is present mostly as inclusions in the quartz.

DISCUSSION

In an effort to determine how well the procedure purifies the quartz for *in-situ*-produced ¹⁰Be and ²⁶Al measurements, these nuclides were determined in material from various steps. The results are shown in Table 3. The sample studied is one of a suite of samples with a known cosmic ray exposure age which was collected for ¹⁰Be-²⁶Al calibration purposes (NI-SHIIZUMI et al., 1989) and which is used here to verify the chemical processing. It is a granitic rock with a moderately coarse crystalline structure and a low intrinsic Al content in the quartz phase. Blanks in the table indicate samples where the stable Al content was too high for the ²⁶Al to be measured.

TABLE 3. Measured ¹⁰Be and ²⁶Al Concentration in Sierra Nevada Granite From Different Chemical Steps*

Step	Weight (g)	Al Concentration	¹⁰ B¢ (10 ⁶ atoms/g)	²⁶ A1 (10 ⁶ atoms/g)	25AI/10Be
Bulk	21.26	7.90 %	71.83 ± 1.58		
After HCl	22.46	6.75 %	11.39 ± 0.32		
After ist ultra	21.31	9960 ppm	1.95 ± 0.09		
After 2nd ultra	40.51	54 ppm	0.50 ± 0.04	2.76 ± 0.23	5.58 ± 0.63
After 3rd ultra	40.32	48 ppm	0.48 ± 0.06	2.82 ± 0.17	588 ± 0.79
After 4th ultra	30.98	56 ppm	0.60 ± 0.10	2.74 ± 0.16	4.58 ± 0.81
 (Nishiizumi e 	(al., 1989)				

The high ¹⁰Be values for the first three steps-grinding (bulk sample), HCl, and the 1st HF-HNO₃ leach—are due to abundant contributions to this nuclide from ¹⁰Be produced in the atmosphere. The low expected concentration of ¹⁰Be produced in situ makes the purified quartz difficult to measure for this nuclide but increases the likelihood of seeing any extraneous activity if it exists in the sample. Indeed, the bulk sample contains over one hundred times the ¹⁰Be measured as an in situ product. This excess represents the accumulation over the rock's 10,000 years' exposure to rain and snow. Once all minerals besides quartz have been removed from the sample, as indicated by the constant and low Al content, this meteoric ¹⁰Be is clearly removed. The agreement among the ¹⁰Be and ²⁶Al data from the second to fourth ultrasonic leaches provides a clear argument that these nuclides are produced in situ in the quartz in the concentrations measured, since subsequent dissolution of surfaces of grains and of the smaller grains with the additional leaches does not change the values measured.

A possible problem with the purification scheme as devised here is that it produced crystals which are heavily etched and have been subjected to considerable heating, which may have leached out some of the *in-situ*-produced nuclides. The evidence above shows that there has been no loss for ¹⁰Be or ²⁶Al. In addition, ²¹Ne has been measured in quartz separated by the HF-HNO₃ leach and in hand-picked quartz. Although the evidence is not conclusive, there is no sign of significant loss of ²¹Ne (G. B. Hudson and J. Poths, pers. commun.). An additional advantage of the etching provided by our procedure is that it removes at least a few microns of the surfaces of the grains and effectively eliminates recoil effects, such as ²¹Ne produced by ²⁴Mg(n, α)²¹Ne from adjacent minerals and ¹⁸O(α , n)²¹Ne from any adjacent high U phases.

AI AND Be SEPARATIONS FROM QUARTZ

Although this paper describes the development of the quartz separation, a brief description of the chemical procedures for separation of Al and Be from quartz for AMS measurements is given below. After the purified quartz is weighed, 0.5–1.5 mg of Be carrier are added to the sample, which is then dissolved with an HF-HNO₃ mixture in a Teflon beaker. The Be carrier was prepared by extracting Be from beryl which was collected from a deep mine, since commercial Be reagent contains a significant amount of ¹⁰Be (1–3 × 10⁻¹⁴)¹⁰Be/Be) (i.e., MIDDLETON et al., 1984). After complete dissolution of the quartz, SiF₄ is evaporated off with HClO₄. Any trace amount of fluoride is eliminated by fuming with HClO₄ three times. The residue is dissolved in HCl and an aliquot taken for Al analysis. If the total amount of Al is less than \sim 1 mg, appropriate amounts of Al carrier are added.

The main solution is run through a 5-mL anion exchange column (MCI CA08P) with 10 N HCl. This step separates Fe and part of the Ti from the Be and Al in the sample, 0.5mL of acetylacetone is added to the elutant, and the solution is neutralized (pH \sim 6) with NH₄(aq.). Aluminum and Be are extracted into CCl4 and then backextracted with an HCl solution. Aluminum and Be are separated from each other using a 5-mL cation exchange column (AG 50W-X8). Beryllium is eluted with 1 N HCl, and Al is eluted with 3 N HCl. The Be fraction is treated with aqua regia and HClO₄ to decompose any trace organic matter. Al(OH)₃ and $Be(OH)_2$ are precipitated with $NH_4(aq.)$ in centrifuge tubes. After the precipitate is washed, the hydroxide is ignited to oxide (Al₂O₃ and BeO) in quartz vials at \sim 950°C. If the original quartz sample was very clean, the anion exchange column or/and acetylacetone extraction steps are eliminated. However, if the quartz contains large amounts of AI or other elements, some modifications are required, such as changing the column sizes or adding a solvent extraction step using EDTA. The procedures described above represent the simplified chemical separation scheme which we have been using for meteorites and lunar samples for a long time (e.g., NI-SHIIZUMI et al., 1984a, 1984b).

SUMMARY OF PROCEDURE

The quartz separation procedure developed from the studies presented here is described as follows. As a first step, the rocks or soils are crushed and ground as necessary to obtain a particle size giving mostly mono-minerallic grains. In most cases, this was $\leq 700 \ \mu$ m. Next, the ground sample is heated in 1:1 HCl with $\sim 0.03\%$ of H₂O₂ (~ 1 g sample per 10 mL). This treatment dissolves carbonates and iron oxides. The residue is washed several times with deionized H₂O, and fines are discarded with the rinse water.

The HCl-treated powder is leached with a dilute HF-HNO₃ mixture to dissolve clays, feldspars, and other silicate minerals. 7.5 g of sample are added per liter of 1% HF-1% HNO₃. Leaches consist of agitating the sample-acid mixture for 9 h in an ultrasonic bath heated to \sim 95°C. Leachant is discarded, then samples are rinsed several times with deionized H₂O and dried. Fines are discarded with the rinse water. This acid leach is repeated as necessary to reduce the Al content in the quartz, a total of two to four times. The amount of acid per gram of sample is reduced by a factor of 2 to 3 for the second to fourth leaches.

The assay for quartz purity is Al analysis performed by atomic absorption spectrophotometry.

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