

# Luxel

# Thermal Evaporation Source RADAK<sup>®</sup> and OLED Models I, II, & III



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# Introduction and Safety Warnings

Thank you for your purchase of a Luxel vacuum evaporation furnace! Your RADAK<sup>®</sup> furnace has been engineered to be a robust, versatile, and affordable source for a wide variety of thermal evaporation applications. Due to high operational temperatures, it is critical for operators to have appropriate training and adhere to all safety recommendations in this manual. Failure to comply with all safety precautions violates the intended use of the furnace and increases the risk of equipment damage and/or personal injury. Numerous installation configurations are possible, with unique needs for safety interlocks, source separation, and thermal shielding. Follow all guidance in this manual, if you have further questions regarding installation and operation of RADAK<sup>®</sup> furnaces please contact Luxel.

# **CAUTION:**

This manual contains important safety information. Critical safety notes are highlighted in CAUTION boxes throughout the manual.

Luxel Corporation assumes no responsibility for damages resulting from the misuse of RADAK<sup>®</sup> vacuum furnaces. Any furnace modifications made without Luxel's written consent will result in termination of the warranty.



# **Quick-Start Guide**

1. Mount furnace securely in system using the ¼-20 threaded mounting boss on the furnace base.



- 2. Connect power lead wires to power terminals, do not short lead wires to furnace body.
- 3. Connect thermocouple lead wires to ceramic plug (observe polarity), insert plug in furnace.



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- 4. Remove furnace outer cover and insert filled crucible gently until it rests on the thermocouple post inside.
- 5. Reinstall cover, ensure crucible is centered with vapor shield opening at the top. Lipped crucibles should have their lip protruding outside the vapor shield, forming a vapor seal.





6. Connect a power supply (40V, 40A is sufficient for most applications). For initial operation, apply power manually and verify source heating.

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# **1. Description and Specifications**

# 1.1 Unpacking

Remove the evaporation source and components from the shipping container. Check the components against the packing list, and notify Luxel of any discrepancy between the parts list and the actual parts received. Inspect all components for shipping damage, and verify the furnace is in good working order before discarding the packaging. If you discover that the furnace has been damaged in shipment, please report the damage immediately to Luxel and the shipping carrier.

The standard items included with a RADAK<sup>®</sup> furnace are:

- Furnace base unit with terminal connections
- Radiation baffle (pre-installed)\*
- Furnace cover (pre-installed)
- One alumina crucible (pre-installed)
- One ceramic thermocouple plug, male end
- One length of positive thermocouple lead wire
- One length of negative thermocouple lead wire
- User Manual

\*NOTE: Luxel OLED furnaces include all the same items as RADAK<sup>®</sup> furnaces with the exception of the radiation baffle, which is not required for temperatures below 600°C.



Figure 1: Standard components of a RADAK<sup>®</sup> furnace include base, radiation baffle, cover, and alumina crucible

If you purchased additional crucibles, liners, or other optional accessories please check these against the packing list.



# 1.2 **Product Description**

Your furnace belongs to Luxel's RADAK<sup>®</sup> line of thermal evaporation sources which can accommodate a wide range of deposition materials. By selecting the proper combination of crucible, crucible liner, thermocouple type, and other options, RADAK<sup>®</sup> furnaces are an ideal source for applications ranging from low temperature organics (OLEDs) to salts and metals used in thin film battery and solar cell production.

# Features:

- Accurately control temperatures to 1500°C with baffle installed (600°C without baffle).
- Internally mounted thermocouple monitors source temperature without exposure to vapors.
- Rapid source material change with no cross-contamination.
- Crucible configuration results in repeatable and reliable deposition.
- Easy access to crucible for cleaning and source replenishment.
- Thermally shielded to minimize chamber heating.
- Easily controlled using low voltage power supply.
- Easy installation for new system design or retrofitting existing systems.
- Can be run in clusters for co-deposition.
- Compatible with commercially available deposition rate controllers.
- Maximizes source material utilization by directing vapor flux to the substrate.

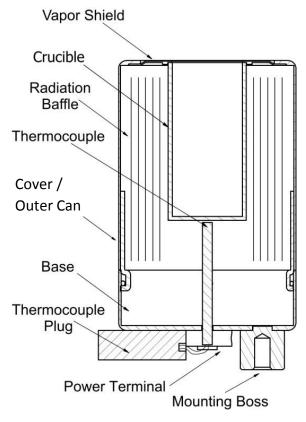


Figure 2: RADAK<sup>®</sup> furnace component names.



# 1.3 Specifications

Table 1:  $RADAK^{\circledast}$  and OLED Furnace Specifications

	OLED I	OLED II	OLED III	RADAK <sup>®</sup> I	RADAK <sup>®</sup> II	RADAK <sup>®</sup> III
Crucible Charge Capacity, cc	1	10	100	1	10	100
Temperature Range, °C	600	600	600	1500	1500	1500
Height, inches (incl. mount boss)	2.60	3.87	6.50	2.60	3.87	6.50
Diameter, inches	1.75	2.25	3.50	1.75	2.25	3.50
Weight, lbs	0.37	0.69	2.44	0.43	0.84	2.85
Operating Pressure, Torr	<5.0x10 <sup>-4</sup> (inert atmosphere)					
Heater Filament, $\Omega$ (±0.05)	0.28	0.35	0.45	0.28	0.35	0.45
Type-K Thermocouple, $\Omega$ (±0.05)	1.35	1.45	0.55	1.35	1.45	0.55
Type-C Thermocouple, $\Omega$ (±0.05)	0.6	0.65	0.9	0.6	0.65	0.9
Typical Current Requirement, A	6 11 18		9	16	18	
Typical Voltage Requirement, V	9	23	50	18	35	50
Maximum Current, A	15	30	40	15	30	40
Maximum Voltage, V (AC or DC)	<b>C)</b> 40 60 40 60			50		



# 2. Installation

### 2.1 Installation Requirements

The RADAK<sup>®</sup> furnace has been designed to function in any position. For evaporative use, mount may vary from vertical to horizontal as may be required for vapor distribution and retention of the source material. Downward evaporation of some material is possible through a special crucible insert. Luxel can assist you in designing a screen to retain subliming materials for horizontal evaporation.

RADAK<sup>®</sup> furnaces have low voltage requirements compared to most evaporative sources. A 30A/40 VAC power supply is adequate for most applications. The furnace is also compatible with a 110 VAC source with a 4:1 step down transformer.

#### Feedthroughs

- 20, 30, or 40 Amp power feedthrough for models I, II, or III respectively
- Thermocouple feedthrough (Type-C or -K depending on furnace configuration)
- (Optional) Rotary motion feedthrough for shutter

#### **Additional Components**

- Bare #10-AWG solid copper wire for connecting power feedthroughs to furnace
- A power supply / temperature controller for controlling source temperature. For automated PID temperature control with programmable ramps and soaks, Luxel's Power Controller II series is recommended. For co-deposition with 2 thermal evaporation sources, Luxel's DUAL Controller is recommended.
- (Optional) A deposition controller for monitoring evaporation rate. Luxel's Power Controllers can accept analog inputs from many common deposition controllers and rate monitors for completely automated deposition with PID control.

#### **Optional Assembly Components**

- A shutter assembly, comprised of a rotary feedthrough, shutter arm, and shutter. Please contact Luxel for assistance with design and ordering options.
- Conflat flange mounted furnaces are available complete with power, thermocouple, and rotary feedthroughs with shutter assembly. Contact Luxel for details.

Because feedthroughs, shutters, and mount configurations depend on the geometry and ports available in your vacuum system, Luxel does not supply a standard mounting flange. Luxel engineers can assist with specifying the hardware appropriate for your system and our shop is available to manufacture custom hardware as needed.

### 2.2 Installation Procedure

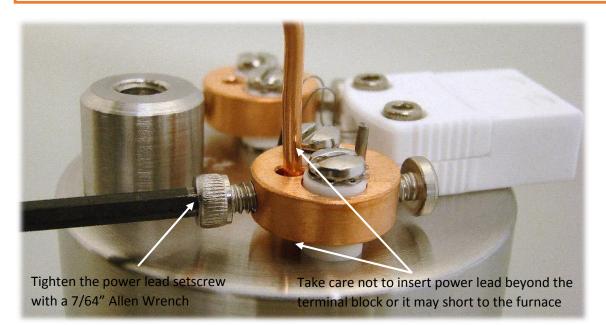
 Bolt the furnace in place using a ¼ -20 bolt in the mount coupling on the base. It should be mounted firmly enough so that the furnace orientation does not change when attaching and removing the cover, this assures a consistent vapor distribution pattern. Excessive torque on the coupling should be avoided.



2. Connect power lead wires to the terminals on the furnace base. The power connection between vacuum system feedthroughs and the furnace may be made with 10 AWG copper lead wire (Figure 2). Insert copper wire ends into the power terminals on the base of the furnace and tighten the socket head setscrews. The setscrews on the side of the power terminals require a 7/64" Allen wrench.

# **CAUTION:**

When inserting the power lead, take care to ensure the power lead does not extend far enough through the power terminal to make contact with the furnace body. This can result in a short to ground through the vacuum system.



#### Figure 3: Power terminal connection.

3. Each RADAK<sup>®</sup> furnace is supplied with thermocouple lead wires and a male ceramic thermocouple plug which provides quick and easy connection to the furnace. Connect thermocouple lead wires to the ceramic plug and the vacuum system feedthrough, ensure that the positive lead wire connects to the positive terminal and that the negative wire is connected to the negative terminal. Proper thermocouple polarity can be verified by directing a heat source such as a lamp or hot air gun on the thermocouple and observing a corresponding temperature increase on the attached controller readout. Insert the male ceramic plug into the connector on the base of the furnace.

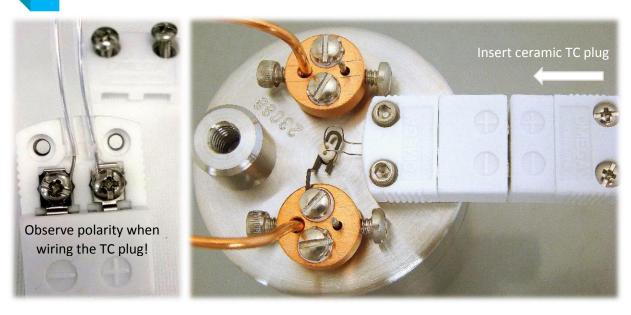


Figure 4: Install thermocouple lead wires in ceramic plug (left). Insert thermocouple plug on furnace base (right).

# **CAUTION:**

Do not allow the thermocouple lead wires to touch the furnace base, power lead wires, or each other. Improper thermocouple wiring can result in furnace over-temperature and can render the furnace unreliable or inoperable. OBSERVE PROPER THERMOCUPLE POLARITY: If thermocouple wire polarity is reversed, you may observe the temperature response to be the opposite of the expected behavior (temperature goes down when power is applied)

4. Verify proper installation by performing the continuity test in Table 2. This may be done outside of the vacuum chamber at the feedthroughs, before connecting the power controller.

Continuity Measurement	Correct reading (Ω)
Power lead to system (ground)	∞
Thermocouple to system (ground)	∞
Power lead to thermocouple	∞
Power lead to power lead (heater resistance)	0.2-0.5 (see Table 1)
Thermocouple (+) to thermocouple (-)	0.6-1.5 (see Table 1)

Table 2: Continuity test values

5. It is the responsibility of the user to ensure appropriate interlocks are installed. For example, power controllers should provide for shutdown on thermocouple failure to avoid thermal runaway.

# 2.3 Installing/Removing the Crucible

Always run the furnace with a crucible installed; the crucible is necessary for temperature measurement. To remove or replace the crucible, follow the steps shown in Figure 5: Remove the outer



furnace cover by twisting counterclockwise (1) and lifting (2). Center the crucible in the furnace and insert gently until it's supported by the thermocouple (3), or to remove the crucible pull it straight up. Reinstall the furnace cover by aligning with the base and gently pressing down (4), then twist clockwise to engage the locking tines (5). When replacing the cover, check that the crucible is centered with the vapor shield to effect a proper seal against vapors entering the furnace. Lipped crucibles (available in a variety of materials) have a lip at the top which locates to the vapor shield and extends slightly beyond it to provide a vapor seal (Figure 6). The crucibles are supported on a spring-loaded thermocouple and the crucible height is critical for making this seal. Crucibles with chipped or broken edges should be discarded.

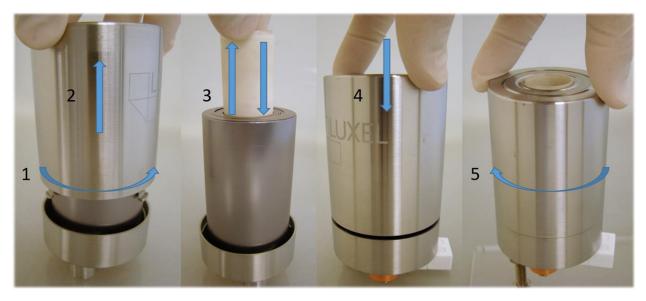


Figure 5: Crucible installation / removal



Figure 6: For lipped crucibles, ensure that the lip protrudes from the vapor shield outside the furnace, indicating the crucible is centered and the vapor seal has been properly formed.

# **CAUTION:**

Make sure that the crucible is centered with the molybdenum vapor shield to effect a proper seal against vapors entering the furnace. Vapors can cause damage to furnace internals, especially the tungsten filament.

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# 2.4 Installing/Removing the Radiation Baffle

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To reach higher temperatures efficiently and minimize heat load to the deposition chamber, RADAK<sup>®</sup> furnaces are fitted with a multi-layer refractory metal shield called the radiation baffle. Because of the radiation baffle's efficient reduction of radiant heat loss, temperature control of the furnace becomes progressively more difficult as the temperature setpoint is reduced below 500°C. While not required, Luxel recommends removing the radiation baffle to deposit materials below 500-600°C (such as OLED source material) since this will facilitate more rapid cooling and better temperature control.

Note: the radiation baffle is easily dented; handle with care. Most denting is cosmetic, however excessive denting can reduce the insulating effectiveness of the baffle.

To remove or replace the baffle, follow the steps shown in Figure 7: Remove the outer furnace cover by twisting counterclockwise (1) and lifting (2). Install the radiation baffle by **gently** sliding the baffle over the heater cage until it rests on the furnace base (3). To remove the baffle, gently pull it straight out of the furnace, do not use excessive force or the heater cage could be damaged. If the baffle gets stuck due to vapor buildup inside the furnace (improper crucible seal) try gently rotating to free it, taking care not to put excessive torque on the heater cage. Reinstall the furnace cover by aligning with the base and gently pressing down (4), then twist clockwise to engage the locking tines (5). When replacing the cover, check that the crucible is centered with the molybdenum vapor shield to effect a proper seal against vapors entering the furnace.

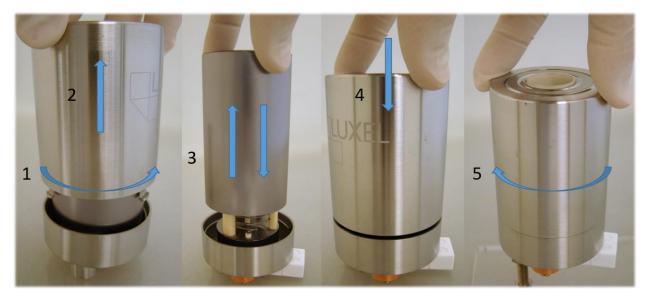


Figure 7: Radiation baffle installation / removal

# **CAUTION:**

The radiation baffle should be easy to remove. If you have any difficulty removing the baffle, proceed with caution to avoid damaging the heater cage.

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# **3.** Crucible and Source Material Considerations

# 3.1 Crucible Operating Temperature

Luxel offers a selection of crucibles and liners compatible with various source materials (for a complete list see Section 9). Always be sure to observe the maximum rated temperatures for these crucibles and liners in Table 3.

Maximum Operating Temperature (°C)					
Crucibles					
Alumina	1500				
PBN	1400				
Quartz	1000				
Coated Stainless	900				
Organic	460				
Liners					
Molybdenum	1500				
Tantalum	1500				
Tungsten	1500				
Nickel	1200				
PBN (Flanged)	1400				

Table 3: Maximum crucible operating temperature

# **CAUTION:**

Operating a crucible or liner above the temperature limits in Table 3 can result in crucible deformation, melting, or cracking causing damage the furnace and voiding the warranty.

**Special note for quartz crucibles:** There is a significant difference in response between quartz crucibles and alumina crucibles. The quartz crucibles are nominally transparent to the IR radiation emitted by the heating elements where the alumina absorbs somewhat better, especially in the lower temperature ranges. The result is the quartz crucible will be at a much lower temperature than the alumina for the same power. The quartz crucible is transmitting the IR from the heating elements directly to the source material so temperature readings will be significantly lower for the same deposition rate when running a material out of a quartz crucible instead of the alumina crucible.

# 3.2 Crucible Selection

Care should be taken to ensure compatibility between the crucible and the material being deposited. Luxel produces a line of crucibles and liners to accommodate a wide variety of source materials; refer to Table 4 and Luxel's downloadable <u>Crucible Selection Guide</u> to choose a crucible for your deposition material. Please note some materials require the use of a liner inside the crucible, as noted in the table.



Table 4: Crucible Recommendation by Material

Material	Symbol	Crucible	Liner	Melt °C	S/D	NOTES
ALQ3	ALQ3	Organic				
Antimony	Sb	Alumina		631	S	0,T
Arsenic	As	Alumina		814	S	0,T
Barium	Ва	Alumina	Та	717		O,P,T
Barium Chloride	BaCl2	Alumina	Ta, Mo	962		
Beryllium	Ве	Alumina	W	1284		
Bismuth	Bi	Alumina	W, Ta, Mo	271		Т
Boron Nitride	BN	Alumina		2973	S	
Cadmium	Cd	Alumina		321	S	P,T
Calcium	Ca	Alumina		810	S	0
Calcium Fluoride	CaF2	Alumina	Mo, Ta	1418		
Cerium	Ce	Alumina		785		Н
Cesium Iodide	Csl	Alumina		621		
Chromium	Cr	Alumina	W	1857	S	
Cobalt	Со	Alumina		1478		
Copper	Cu	Alumina	Мо	1083		
Copper Phthalocyanine	CuPC	Organic				
Dysprosium	Dy	Alumina	Та	1409		
Erbium	Er	Alumina	Ta, W	1522	S	
Europium	Eu	Alumina		822	S	0
Gadolinium	Gd	Alumina		1312		
Gallium	Ga	Alumina	PBN	30		
Germanium	Ge	Alumina	PBN	959		
Gold	Au	Alumina		1063		
Holmium	Но	Alumina	Та	1470		
Indium	In	Alumina		157		
Indium Tin Oxide	ITO	Alumina		1800	S	
Iron	Fe	Alumina		1535		
Lanthanum	La	Alumina	Ta, W	887		0
Lead	Pb	Alumina		328		Т
Lutetium	Lu	Alumina		1656		
Magnesium	Mg	Alumina		651	S	0
Magnesium Fluoride	MgF2	Alumina		1261		
Manganese	Mn	Alumina		1244	S	
Neodymium	Nd	Alumina		1021		
Nickel	Ni	Alumina		1455		
Palladium	Pd	Alumina		1555	S	
Phosphorus	Р	Alumina		597	S	T,F

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Material	Symbol	Crucible	Liner	Melt °C	S/D	NOTES
Polonium	Ро	Quartz		254		Т
Potassium	К	Quartz		64		F
Potassium Bromide	KBr	Alumina		734		
Potassium Chloride	ксі	Alumina	Ni	770	S	
Potassium Iodide	KI	Alumina		681		
Praseodymium	Pr	Alumina	Та	931		
Rubidium	Rb	Quartz		38		F
Samarium	Sm	Alumina		1072		0
Scandium	Sc	Alumina		1397		0
Selenium	Se	Coated SS		217		Т
Silicon	Si	Alumina	Та	1410		
Silicon Monoxide	SiO	Alumina	Та	1702	S	
Silicon Nitride	Si3N4	Alumina		1900		
Silver	Ag	PBN, Alumina	Мо	961		
Sodium	Na	Quartz		98		F
Sodium Fluoride	NaF	Alumina		993		
Strontium	Sr	Alumina	Mo, Ta, W	771		O,F
Sulfur	S	Quartz		597	S	Р
Tellurium	Те	Alumina	Ta <i>,</i> W	450		Т
Terbium	Tb	Alumina		1360		
Thallium	TI	Alumina		304		Т
Thullium	Tm	Alumina		1545	S	
Tin	Sn	Alumina	Та	232		
Tin Oxide	SnO2	Alumina		1630	S	
Titanium Oxide	TiO	Alumina	W, Mo			
Tungsten Oxide	WO3	Alumina		1473	S	
Ytterbium	Yb	Alumina	Та	824	S	
Yttrium	Y	Alumina		1477		
Zinc	Zn	Alumina		419	S	Р

#### NOTES:

H = Hydroscopic films

O = Oxidizes quickly in air

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P = Poisonous to vacuum systems due to low sticking

coefficient

- T = Toxic
- F = Flammable in air
- S = Sublimes
- D = Decomposes



# 3.3 Crucible Filling

The appropriate crucible fill level depends on the material being deposited.

**Subliming Materials:** The furnace may be operated with the crucible nearly full of subliming materials in the form of pellets or chunks. During an evaporation at constant power or temperature, the vapor distribution and rate will change with depletion. Few problems have been encountered with materials that sublime or materials with little structural strength, such as most salts.

**Non-Subliming / Molten Materials:** The maximum recommended crucible fill level for nonsubliming materials is ¾ full, resulting in a usable capacity of 1cc, 10cc, and 100cc for RADAK<sup>®</sup> models I, II, and III respectively (Figure 8). Special care must be taken when evaporating molten metals. Molten metals may be evaporated but if the residual charge is too large, the alumina crucible may be cracked upon reheating due to the difference in expansion coefficients. Luxel produces a line of crucible liners for molten metal evaporation and we encourage you to use one.



Figure 8: Recommended crucible fill level for non-subliming materials is 3/4 full.

**Chemical compounds:** Best results are achieved with the crucible about half-full and depletions down to about one sixth full. Evaporations where vapor qualities such as dissociation, varying rate, or vapor temperature are not a factor can be carried to completion without any other consideration.

# **CAUTION:**

Failure to use the proper crucible/liner combination with your material may result in leaking molten material into the furnace body, destroying the furnace. For molten materials Luxel recommends inspecting the crucible for signs of cracking after each melt & cool-down cycle.



# 4. Operation

### 4.1 Heater Control

The furnace is resistance-heated and may be powered with a 30A/40 VAC power supply, or a 110 VAC source with a 4:1 step down transformer. Very stable temperature and deposition rates can be achieved with PID heater control. Luxel manufactures a line of power controllers to accommodate both sequential deposition and co-deposition processes with flexible automation options. Contact Luxel for a recommendation based on your process, or go to <u>luxel.com/products/thermal-evaporation-</u><u>equipment/power-controllers</u> for further details.

# 4.2 **Operating Environment**

The user should be aware of the following guidelines for proper operation of the vacuum furnace.

#### **Partial Pressures**

The furnace core and thermal insulation are comprised of tungsten, tantalum and alumina. Exposure of these materials to certain gases or vapors at high partial pressures while the furnace is at high temperature may degrade and shorten the life of these components. These gases and vapors are most notably, but not limited to, nitrogen, oxygen and water vapor. RADAK® furnaces should be operated in an inert atmosphere of <5.0x10<sup>-4</sup> Torr vacuum or better. Luxel recommends <5.0x10<sup>-6</sup> Torr for typical applications. Higher operating pressures may be possible depending on the inert gas environment and furnace temperature but this will void the warranty. Note that heat load to the chamber will increase substantially with operating pressure.

#### **Exposure to Other Sources**

RADAK<sup>®</sup> furnaces are often installed in systems with multiple sources, placed closely together. In this case, it is important to consider the possibilities of cross-contamination and heat loading from neighboring sources. To mitigate these concerns it is advisable to install some form of shielding to eliminate line of sight between sources and create a thermal barrier that blocks radiative IR heating. For example, a thin sheet of stainless steel mounted between RADAK<sup>®</sup> sources works well.

#### **Furnace Temperature Limits**

RADAK<sup>®</sup> furnaces may be operated to 1500°C with the radiation baffle installed. Without the baffle, operation up to approximately 600°C is possible depending on the power supply. Significantly greater power is required to heat the furnace without the baffle installed due to increased radiative cooling.

# **CAUTION:**

Operating a RADAK<sup>®</sup> above the 1500°C limit may damage some furnace components and void the warranty.



#### **Backfill Temperature Limits**

Portions of the RADAK vacuum furnaces are constructed of tantalum and tungsten metal, which are susceptible to damage by reactive gases. Do not add gases other than noble gases to the vacuum system while the furnace is above 300°C, or these parts will become brittle and weaken.

In regular operation, the furnace should be allowed to **cool below 100°C** before opening the chamber or backfilling with air to avoid oxidizing the filament.

To accelerate cooling and cycle time it is possible to backfill the vacuum chamber once the furnace is below 300°C. Only use nitrogen or a noble gas such as Argon when backfilling the chamber at temperatures greater than 100°C.

# **CAUTION:**

A furnace at 100°C may still be hot enough to burn! Use care when handling the furnace or crucible soon after venting the system.

#### Voltage Limit at High Temperature

Damage may result if RADAK furnaces are operated in excess of 40 volts to ground while at temperature above 1100°C. Ionic conduction through ceramic insulation results in localized heating. When using an AC power supply, control furnace power through an isolation transformer.

### 4.3 Initial Operation

Until connections have been verified through operation, exercise considerable caution during initial commissioning. A few extra minutes now can save hours of avoidable repair.

#### Make Sure a Crucible Is Installed

Always run the furnace with a crucible installed. The crucible can be empty, but it must be installed for temperature measurement.

#### Apply Power Manually for the First Run

The first time you power your furnace, we recommend running in manual mode. In manual mode on Luxel controllers, the operator directly controls the power to the furnace. If using a Luxel Power Controller, a good test power is 10%. If correctly installed, 10% will result in an increase in furnace temperature with no risk of furnace damage. If temperature does not increase, be sure to check the system pressure to rule out any issues with the thermocouple installation. If the temperature decreases, thermocouple polarity is reversed and the thermocouple circuit should be checked and the polarity corrected. Reversed polarity somewhere in the thermocouple circuit is one of the most common issues reported during initial installation; this is the reason for testing in manual mode first. In automatic mode, the controller applies power to reach the temperature setpoint. If the thermocouple wires are reversed and the temperature decreases, the controller will increase power output, causing the indicated temperature to decrease further and so on.



#### **Verify Furnace Heating**

When you first apply power to the furnace, it's advisable to have a second method to verify the furnace is heating. This is especially true for systems with multiple furnaces where crossing thermocouple or power leads could result in a controller powering one furnace but monitoring temperature on another. Two ways to verify the furnace is heating other than the thermocouple indication is visual observation of the crucible and monitoring system pressure. Once the furnace reaches several hundred degrees, a glow should be observable. If the furnace has been exposed to air prior to heating, some moisture will likely remain on the furnace internals despite high vacuum levels in the vacuum chamber. Once the furnace begins to heat, this water is driven off and can be observed via pressure gauge or residual gas analyzer (RGA). The response of the pressure gauge will likely be considerably faster than direct observation.

### 4.4 Routine Inspection

#### **Crucible and Liner**

Inspecting the crucible and furnace between deposition cycles can avoid potential problems leading to expensive furnace damage. For source materials that melt and re-crystallize with each deposition it is especially prudent to check the crucible between cycles. If source material has spilled out of a liner into the crucible due to cracking or wetting over the rim of the liner, replace both the crucible and liner. The RADAK® furnace has been designed with higher power density near the top of the crucible to avoid vapor condensation at the crucible rim which could wick over the top and damage the furnace, or build up and "choke" the crucible aperture. However, certain materials may still condense near the top of the crucible and cause these issues. If you notice condensation near the rim of the crucible, replace the crucible before material builds up and wets over the edge, or chokes off the crucible opening. Note: for certain materials it may be possible to clean the crucible aperture without replacing the crucible.

#### **Heater Cage**

The heater cage consists of a tungsten filament supported by ceramic insulator posts. Periodically check the ceramic posts for signs of contamination. Luxel now offers a variety of lipped crucibles which provide an effective seal against vapors entering the furnace. However, improper installation of the furnace cover can leave an opening for vapors to enter the furnace. The gradual buildup of conductive film layers on the ceramic insulators leads to shorting of the filament and loss of performance. Clean the furnace if contamination is observed.

The tungsten filament will oxidize and react with gases in the chamber at high partial pressures. Over time this can lead to embrittlement and failure of the filament. Inspect the filament visually: it should be metallic and uniform in color. Any other coloration is likely a sign of oxidation. Make sure you are operating the furnace within the parameters discussed in the section 4.2. If needed, the filament is a user-replaceable component (see section 5.2).



#### 5. Maintenance

# 5.1 Cleaning

The furnace is made of low-outgassing high-temperature materials. The furnace body is stainless steel with a molybdenum vapor shield. The interior is tantalum, molybdenum, tungsten, and high purity alumina. Cleaning, when necessary, should be restricted to the stainless-molybdenum cover and the alumina crucible. Cleaning methods will depend on the material to be removed, but acids, bases, and scouring materials, which do not strongly attack stainless, molybdenum, or alumina can be used. Cleaning of the multi-layer radiation baffle is not recommended beyond ultrasonic cleaning in hot water followed by a rinse in clean solvent such as distilled isopropyl alcohol, and oven drying.

A word of caution about using acids to clean the furnace cover. The molybdenum vapor shield will be quickly attacked by most acids, therefore exposure to acid should be minimized. To remove the brownish oxide created by acid exposure, a quick immersion in H<sub>2</sub>O<sub>2</sub>:NH<sub>4</sub>OH, 1;1, (H<sub>2</sub>O<sub>2</sub> is 35%, NH<sub>4</sub>OH is 28-30% as NH<sub>3</sub>) will work.

An effective, general-purpose cleaning procedure to remove grease and other organics is outlined here:

- Dissolve 2.5 tsp's (1.5 oz) of Alconox<sup>©</sup> detergent in 1 gallon of hot (130°F) water.
- Immerse furnace and outer can in the solution (not the radiation baffle, see note above).
- Place in ultrasonic cleaner for up to 1 hour
- Rinse thoroughly in tap water initially and finish the rinse in DI water.
- Dry components at 60°C for 5-10 min.
- Place components in Acetone and ultrasonic clean for 15 min.
- Place components in Isopropyl Alcohol and ultrasonic clean for another 15 min.
- Dry components at 60°C for 30 minutes or longer if required.

### 5.2 Heater Replacement

In the event that the tungsten heater filament is broken, Luxel offers a heater replacement kit that can be field-installed by the user. To order the correct heater kit, look up the part number for your furnace model in Section 9: Accessories and Order Codes . Detailed instructions for the heater replacement procedure can be downloaded from the <u>RADAK® webpage</u>.



# 6. Troubleshooting

Possible Cause(s)	Solution			
Thermocouple polarity is reversed	Check thermocouple wires at each plug / feedthrough and correct any reversed connections			
Unplugged thermocouple or bad wiring connection	Check that the ceramic thermocouple plug is plugged in. Check continuity of thermocouple wires from furnace plug all the way out to the temperature controller			
Broken thermocouple	Replace thermocouple. This can be done by the user: order new thermocouple from Luxel and follow instructions posted on the website.			
Thermocouple wires in contact with system ground or other wires	Ensure wires are not touching the chamber, themselves, or any other wires. Use insulated thermocouple lead wires if necessary.			
Temperature controller is configured for the wrong thermocouple type	Configure the temperature controller thermocouple type to match the furnace thermocouple type			
Power lead(s) disconnected or shorted	Check that power leads wires are secured tightly to the power terminals on the furnace base (check set screws are tight). Test for short between power leads and system (ground short)			
Heater filament is broken	Disconnect power leads to the furnace and check for continuity between the power terminals. If there is no continuity then the filament may be broken. Install a heater replacement kit.			
Improper seal between crucible and vapor shield resulting in deposition inside the furnace	If the crucible is "welded" to the top ring of the heater cage, it may not be possible to remove it. Attempt to remove the material buildup on the ring. If unsuccessful, return furnace to Luxel for a rebuild.			
Incompatible crucible / material combination resulting in spilled material inside furnace	Spilled material inside the furnace typically results in major damage. Return furnace to Luxel for a rebuild. Note: in extreme cases it may not be possible to rebuild the furnace.			
	Thermocouple polarity is reversed Unplugged thermocouple or bad wiring connection Broken thermocouple Thermocouple wires in contact with system ground or other wires Temperature controller is configured for the wrong thermocouple type Power lead(s) disconnected or shorted Heater filament is broken Heater filament is broken Improper seal between crucible and vapor shield resulting in deposition inside the furnace Incompatible crucible / material combination resulting in spilled			

Radiation baffle is stuck in the furnace	Improper seal between crucible and vapor shield resulting in deposition inside the furnace	The multi-layered radiation baffle is delicate, do not use excessive force to remove it if stuck. Try gently twisting to free the baffle. If this does not work, try cleaning the furnace with the "general purpose" cleaning method before attempting to remove it.
	Radiation baffle is bent or deformed from rough handling / cleaning	Replace the radiation baffle. If it cannot be removed, return furnace for a rebuild.
Furnace unable to reach maximum temperature (1500°C)	Insufficient supply power	Your power supply should be able to provide at least 20 AC Amps to the furnace. Check that an appropriate output limit is set in the controller, and that the temperature setpoint limit is not less than 1500C.
	Incorrect thermocouple reading	Ensure wires are not touching the chamber, themselves, or any other wires. Use insulated thermocouple lead wires if necessary.
	Radiation baffle is not installed	Install the radiation baffle
	Weak thermocouple spring	Test spring action when installing furnace cover. Replace thermocouple spring if there is no spring compression when twisting the cover closed.
Poor PID Control at low temperature	Radiation baffle is preventing radiant heat loss, producing slow cooling response when power is reduced	Remove radiation baffle

*NOTE:* For additional troubleshooting questions and information, please refer to the <u>RADAK® FAQ</u> on our website. If your question is not answered there, please contact Luxel for assistance.

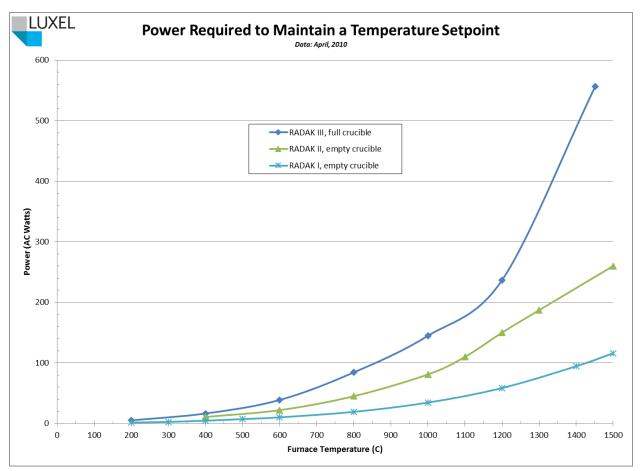


# 7. Performance Curves

The following performance data was collected for RADAK<sup>®</sup> and OLED furnaces loaded with an empty alumina crucible (unless otherwise noted), heating at a ramp rate of 50°C/min using a Luxel Power Controller II. This information is intended for informational purposes only, actual performance values will vary depending on your system setup, deposition material, and crucible charge quantity.

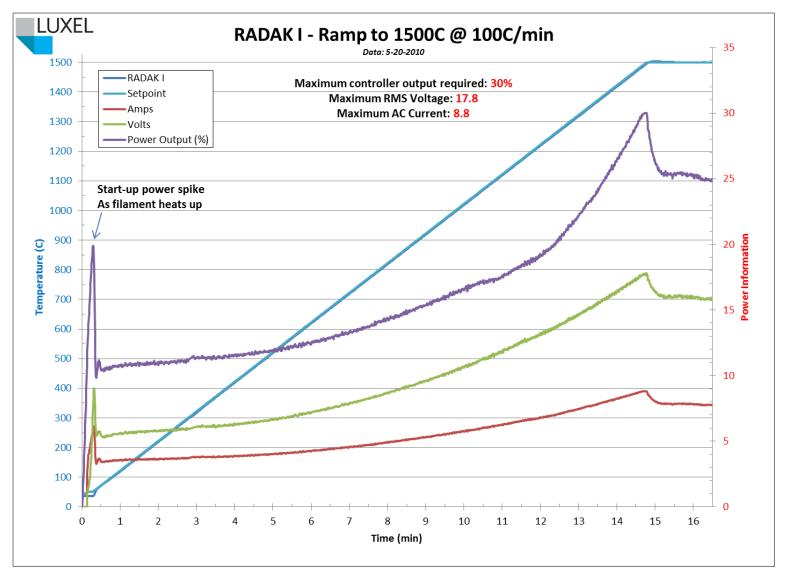
# 7.1 Power to Maintain a Setpoint

The power required to maintain a temperature setpoint for RADAK<sup>®</sup> I, II, and III furnaces is shown below. Note that this power is lower that the power required to maintain a *ramp rate* up to temperature (following sections).





### 7.2 RADAK<sup>®</sup> I Power Curve



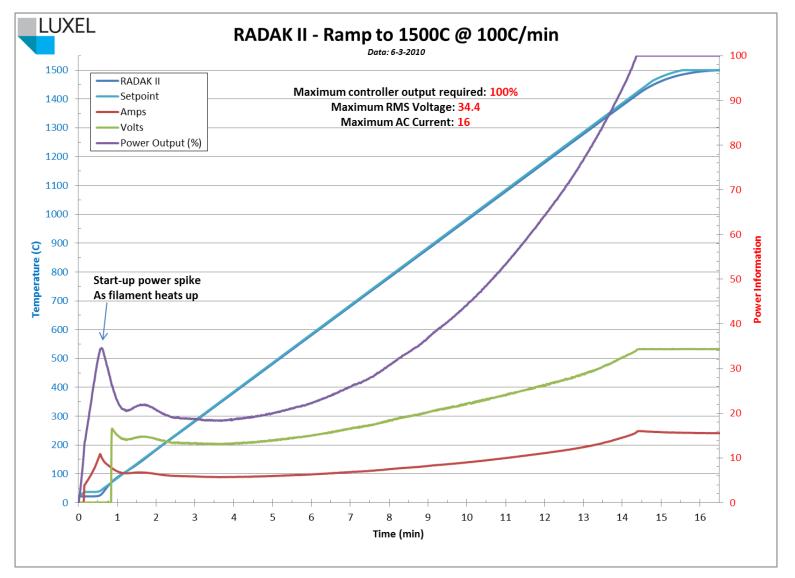
PO Box 1879 60 Saltspring Dr. Friday Harbor, WA 98250 Page 26

Phone: (360) 378-4137 Fax: (360) 378-4266 Email: radak@luxel.com

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### 7.3 RADAK<sup>®</sup> II Power Curve

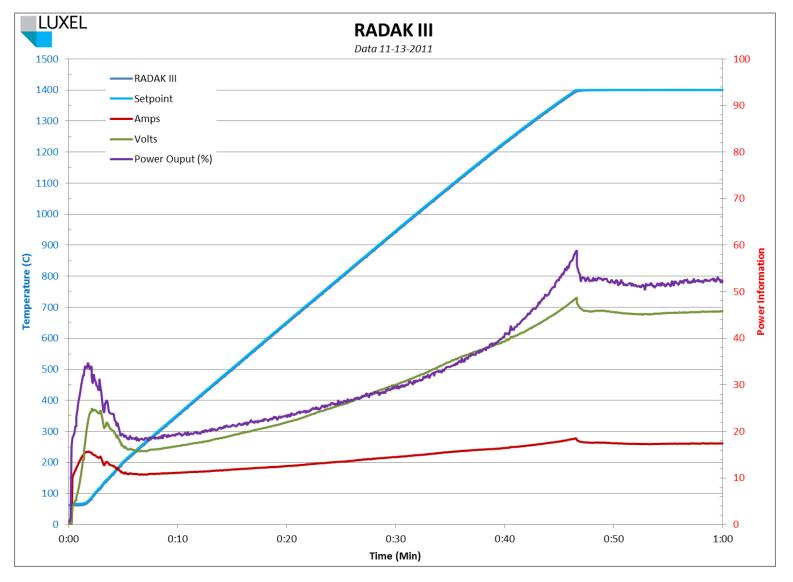


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### 7.4 RADAK<sup>®</sup> III Power Curve

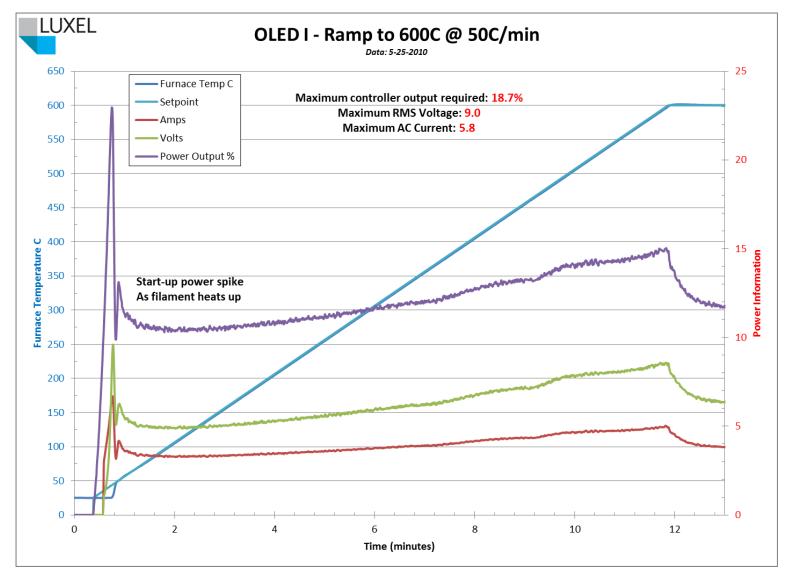


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# 7.5 OLED I Power Curve



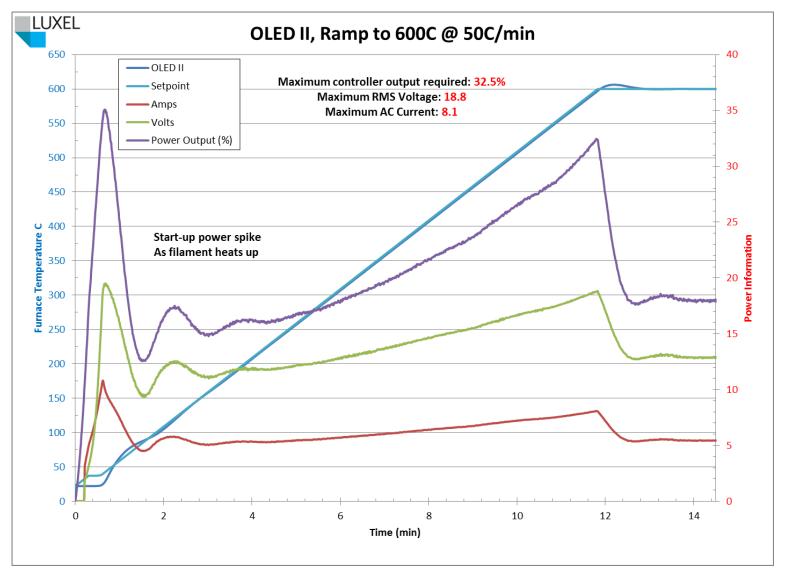
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# 7.6 OLED II Power Curve

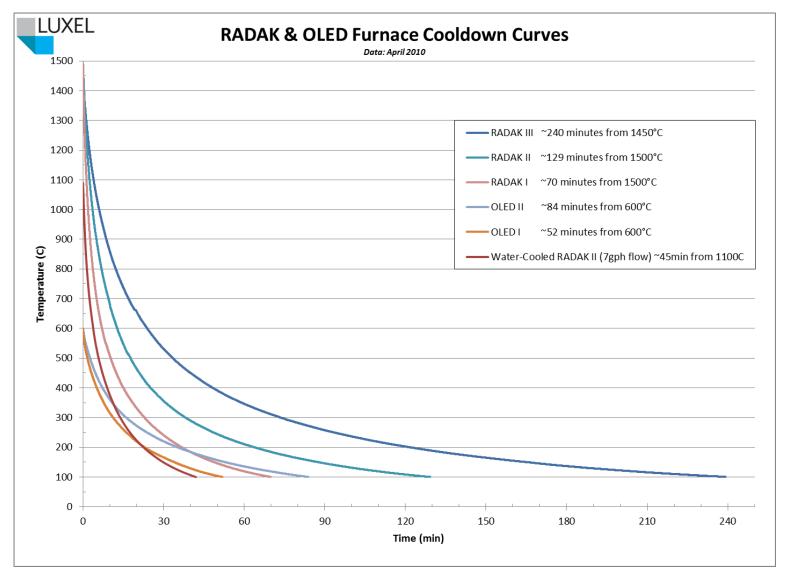


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# 7.7 Cool-down Times



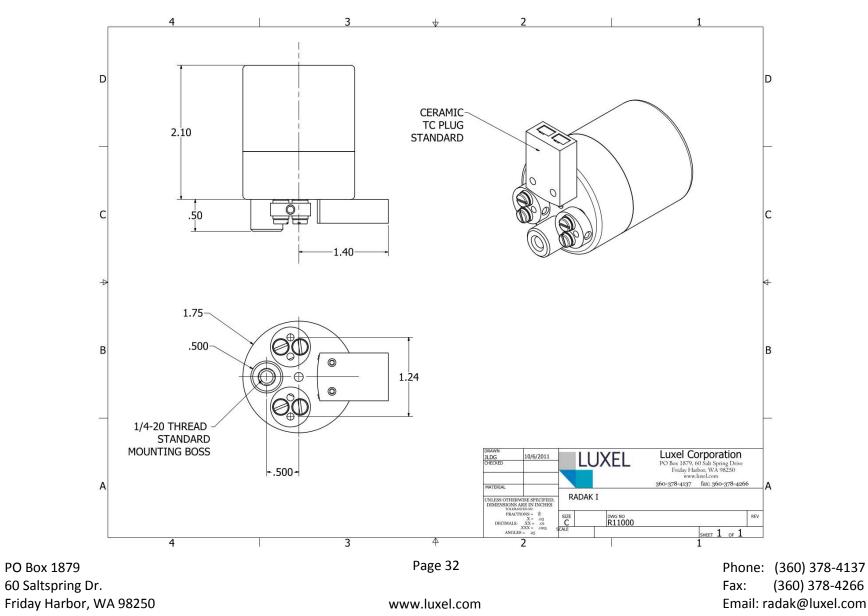
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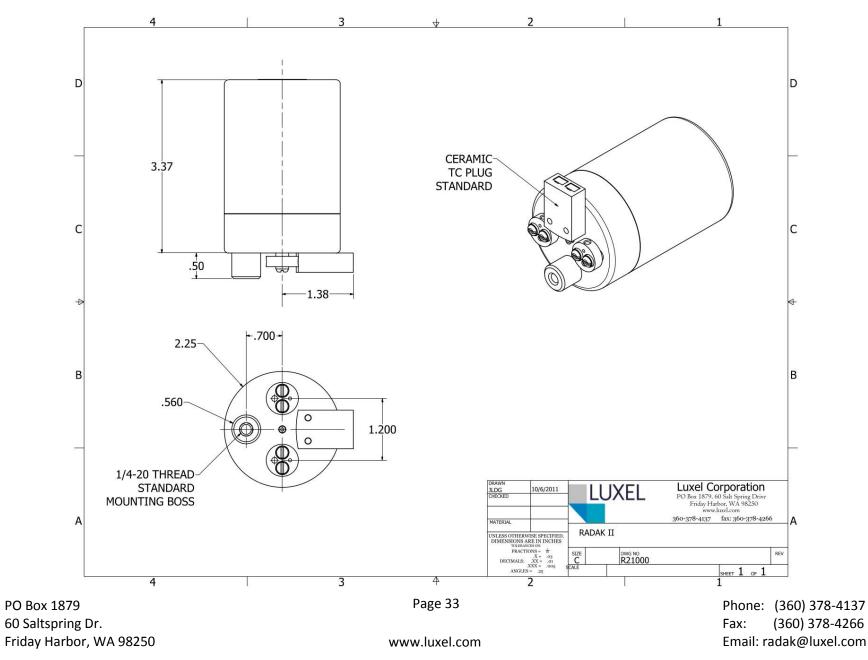
# 8. Drawings

#### 8.1 RADAK I Furnace Drawing



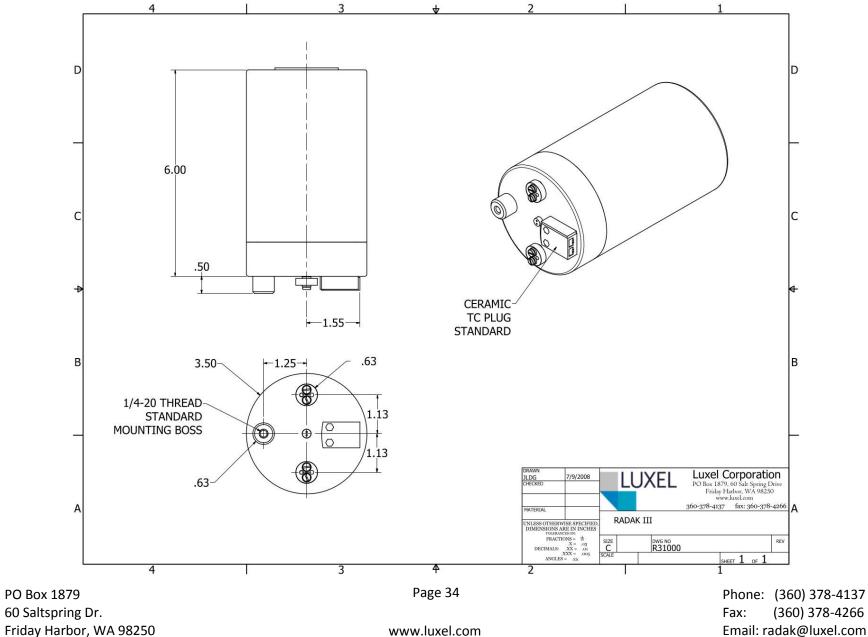
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### 8.2 RADAK II Furnace Drawing



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### 8.3 RADAK III Furnace Drawing





# 9. Accessories and Order Codes

Vacuum Furnaces	Order		
RADAK I Vacuum Furnace, 1cc, 1500°C, Std. Options	11000-C (or -K)		
RADAK II Vacuum Furnace, 10cc, 1500°C, Std. Options	21000-C (or -K)		
RADAK III Vacuum Furnace, 100cc, 1500°C, Std. Options	31000-C (or -K)		
OLED I Vacuum Furnace, 1cc, 600°C, Std. Options	11000-C (or -K)		
OLED II Vacuum Furnace, 10cc, 600°C, Std. Options	21000-C (or -K)		
OLED III Vacuum Furnace, 100cc, 600°C, Std. Options	31000-C (or -K)		
Option w/SS Pwr terminals, Vap. Shld, Arc-Coat SS Crucible	. ,	Irnace model# abov	el
Power Controllers	PART #		0]
RADAK Power Controller //	42010		
RADAK Power Controller //-	42010		
RADAK Power Controller IIP	42015		
RADAK DUAL Power Controller Rack Mount Kit: Dual - 1 Pair of Rack Ears	42100 42021		
	42022		
Rack Mount Kit: Single - 1 Rack Ear, 1 Rack Extension Wire Harness, Controller II+	42015-2		
Wire Harness, Controller IIP	42013-2		
Accessories by Furnace Model:			
	RADAK I	RADAK II	RADAK III
Crucible, Alumina lipped rim	10300-1L	20300-1L	30300-1L
Crucible, Quartz flat rim	10300-2	20300-2	30300-2
Crucible, PBN flat rim (single walled)	10300-3S	20300-3S	30300-3S
Crucible, Organic, lipped rim (temp limit 450C)	10300-4	20300-4	30300-4
Crucible, Arc Coated SS, lipped rim-RI; reduced aperture-RII & RIII	10300-5	20300-5	30300-5
Crucible Liner, Tungsten	10301-1	20301-1	30301-1T*
Crucible Liner, Molybdenum (tall)	10301-2T	20301-2T+	30301-2T
Crucible Liner, Tantalum (short)	10301-3	20301-3	30301-3*
Crucible Liner, Nickel (short)	10301-6	20301-6	30301-6*
Crucible Liner, PBN (single walled)	10301-8*	20301-8	30301-8*
Crucible Liner, PBN w/Flanged rim (single walled)	10301-8F	20301-8F	30301-8F
Thermocouple, Type C, W:5Re/W:26Re	10411-C	20411-C	30411-C
Thermocouple, Type K, Chromel/Alumel	10411-K	20411-K	30411-K
Heater Replacement Kit	10412	20412	30412
Clean Shields, 5/pkg. Flat Top (Stainless Steel)	10105-2	20105-2	30105-2
Vapor Shield, Molybdenum (Standard)	10115-2	20115-2	30115-2
Vapor Shield, Stainless Steel (for Selenium)	10117-2	20117-2	30117-2
T/C bracket, 90° - to mount plug in vertical position	10410	20410	30410
Power Terminal Universal Mounting Kit	10415-U	20415-U	30415-U
Power Terminals, Set of 2 Copper	10415-CU	20415-CU	30415-CU
Power Terminals, Set of 2 Stainless Steel (for Selenium)	10415-SS	20415-SS	30415-SS
Ceramic Tube insulator, Thermocouple Post	10416	20416	-
Ceramic Tube insulator, Inner, Thermocouple (RADAK III)	-	-	30416-I
Ceramic Sleeve, Outer, Thermocouple (RADAK III)	-	-	30416-O
Ceramic Tube insulator, Set of 2 for Power Feeds	10417	20417	30417
TC Terminal Assembly	10418	20418	30418
Ceramic TC Plug, Type-C, Female, for furnace base	10418-C	20418-C	30418-C
Ceramic TC Plug, Type-K, Female, for furnace base	10418-K	20418-K	30418-K
Ceramic TC Plug, Type-C, Male	10419-C	20419-C	30419-C
Ceramic TC Plug, Type-K, Male	10419-K	20419-K	30419-K
Radiation Baffle	10511-2	20511-2	30511-2
	10511-2 10515-2	20511-2 20515-2	30511-2 30515-2

\*Non-stock item, price on request

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# 10. Warranty and Liability Disclaimer

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Your RADAK<sup>®</sup> vacuum furnace is guaranteed, when used as directed, against defect due to materials or workmanship for 1 year from date of sale with certain reservations. Such reservations are:

- The furnace core and thermal insulation are comprised of tungsten, tantalum and alumina. Exposure of these materials to certain gases or vapors at high partial pressures while high temperatures may degrade and shorten the life of these components. These gases and vapors are most notably, but not limited to, nitrogen, oxygen and water vapor. Such misuse will void this warranty.
- 2. Because the delicate installation is under the control of the user, no warranty is offered with respect to the thermocouple. In particular, no warranty is offered with respect to the tungsten:rhenium (Type C) thermocouple. The best techniques and materials are employed in fabrication of the thermocouple but certain materials, most notably zinc, will open the thermocouple junction for this particular alloy couple. Thermal controllers should provide for shutdown on thermocouple failure. Replacement thermocouples are available at nominal cost; they can be replaced by the user.
- 3. No warranty is offered with respect to crucible damage caused by mechanical or thermal stress due to charge material.

Damaged RADAK furnaces may be returned and inspected at Luxel to determine the nature of the damage. Repairs will be free within the warranty period if the damage is determined to have been caused by manufacturing fault.