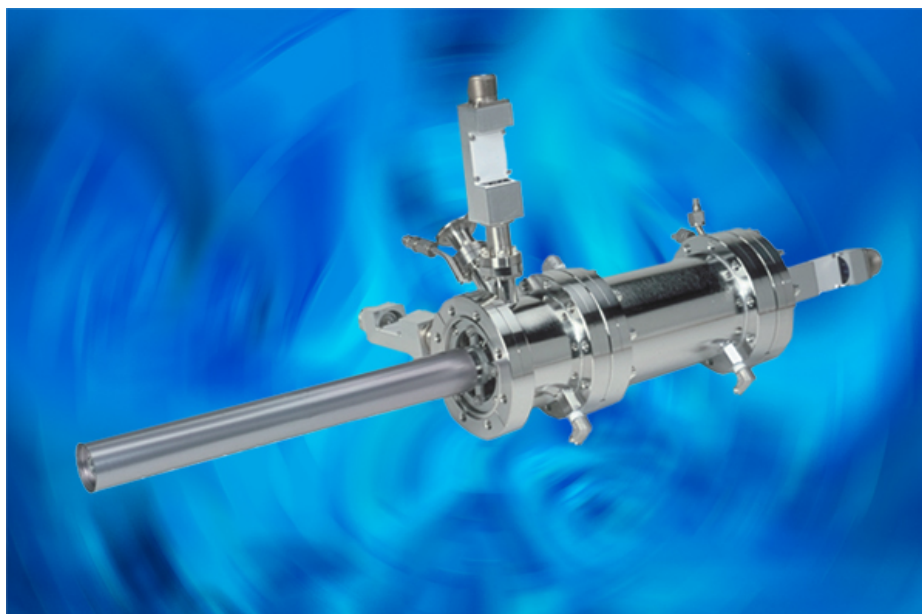


As Valved Cracker Model VC45-As-200 Manual



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Introduction

The SVT Associates' Arsenic Valved Cracker Model VC45-As-200 is designed for operation in a UHV environment. It mounts on a standard 4.50" CF flange and has a capacity of 200 cc. The cracker has a maximum in-system diameter of 1.4 inches and an in-system length of 12 inches. There are heaters and thermocouples in the main body (bulk evaporator/crucible) and the cracking head (tip). All heaters are thoroughly tested and characterized before shipment. Temperature versus power curves for the heaters are included at the end of this manual.

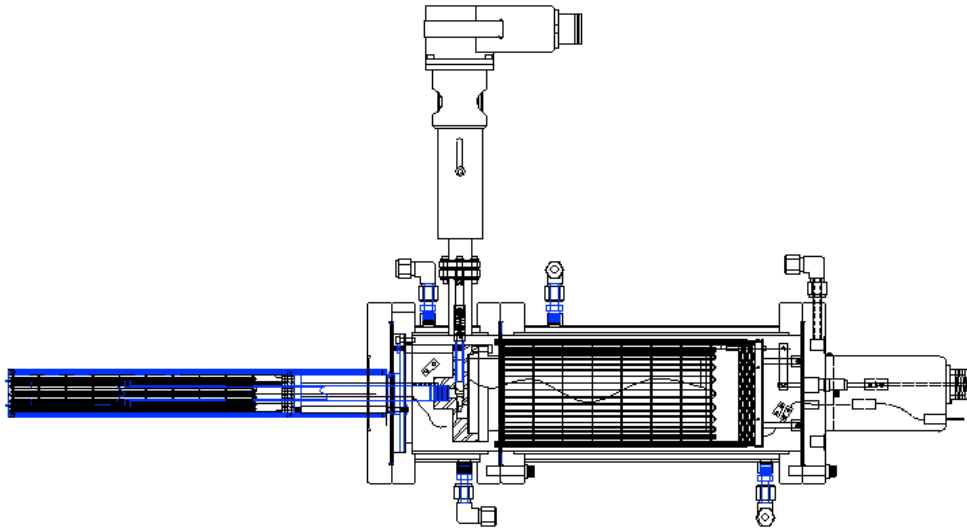


Figure 1: Valved Cracker Source

Unpacking

NOTE: SVT Associates' VC45-As is shipped partially disassembled in several boxes. However, the source was clean and has been tested in a UHV system before shipping. Wear gloves when handling the cracker to avoid contaminating any of the parts which will be in a vacuum.

NOTE: Do not discard any shipping materials until all of the accessory parts (gaskets, bolts, tubing, etc.) have been identified. These are shipped in smaller boxes within the large ones but due to the packing materials they can easily be overlooked.

NOTE: Depending on what options were ordered, there may be several boxes containing electronic equipment and cables. The cables may not necessarily be shipped in the same box as their corresponding pieces of equipment.

The Valved Cracker

Carefully remove and unpack the four main sections of the cracker:

1. **Cracking head:** This includes the cracking (tip) heater assembly, upper water jacket, valve assembly, linear motion feedthrough valve positioner (with optional stepper motor if ordered) and heater/TC connector block.

Remove the bolts securing the shipping tube to the 4.5" flange. Carefully slide the tube off the heater assembly. Remove the 12-point bolts and washers from the 4.62" flange. Remove the blank flange. Inspect the entire assembly for shipping damage. Save the bolts and washers for later use during assembly.

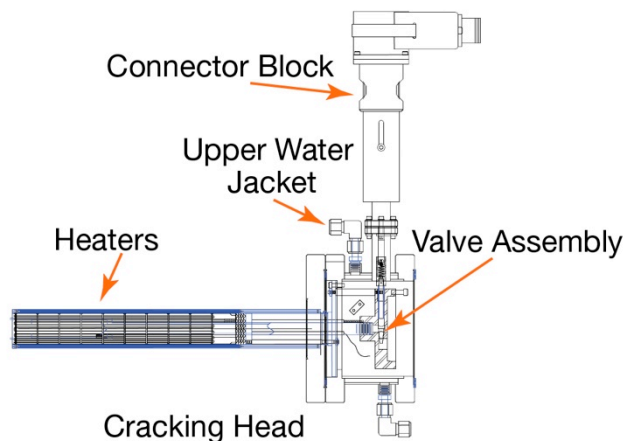


Figure 2: Cracking Head

2. **Bulk evaporator and Lower water jacket:** This includes the bulk heater assembly and its heater/TC connector block. The water jacket fits over the bulk heater assembly and connects the bulk evaporator to the cracking head. It has a 4.62" flange on each end and two water connections. Remove the bolts securing the blank flange. Carefully slide the blank off the heater assembly. Remove the protective cover from the connector block. Inspect for damage. See Figure 3.

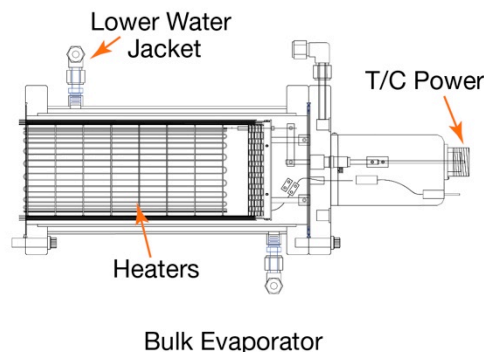


Figure 3: Bulk Evaporator

3. **Molybdenum crucible:** This part will hold the Arsenic liner during operation of the source. It has been heated during testing but has **not** been filled with Arsenic. It mounts on the valve body in the cracking head and fits inside of the bulk heater. See Figure 4.

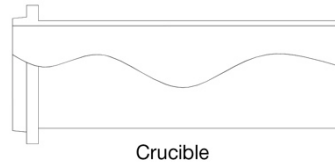


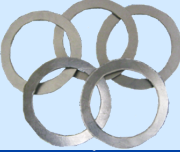



Figure 4: Crucible

Find all the other parts in the various boxes. These include gaskets, bolts, three pieces of water tubing with connectors, manual(s) and possibly software. Inspect each part for shipping damage.

Save the shipping tubes, foam and boxes in case the cell has to be returned for any reason.

Hardware List

Quantity	Size	Description	Image	Part Number
1	200 cc	Molybdenum Crucible		5009242
9	8-32 X 1/2"	Titanium Socket Head Cap Screw		5000434
5	200 cc	Graphoil Crucible Mounting Gasket		5030756
3	6"	Gold Plated Gasket		5009873

Cables

Several cables are shipped with the Cracker, depending on options.

- Two cables (bakeable to 250 °C) for the heaters. These cables have a 2-pin metal circular connector and a ceramic Omega thermocouple connector (attaches to the cracker). Screws and a plastic Omega thermocouple connector for the heater power supplies and temperature controllers.
- Two cables with circular connectors on one end and DB-25 connectors on the other end. These connect from the back of the controller to the three power supplies.

- If the motorized option was ordered, there will be one cable with 10-pin circular connectors on both ends for the motor. Identify and inspect all cables for damage. Refer to the motor controller manual for details.

Valve Motor Control Option

The standard Cracker has a manually operated linear motion feedthrough that controls the valve positioning. If you have ordered the motor control option, a stepper motor will be mounted on the back of the linear motion feedthrough and a limit switch assembly. In addition, the Motor Controller electronics will be installed in the same 19" rack mount chassis as the temperature controllers and power supplies.

Other Options

Any other options that were purchased with Model VC45-AS-200 Valved Cracker such as power supplies, temperature controllers or a computer will be shipped in individual boxes. Unpack them carefully and inspect them for any damage. The temperature controllers are mounted in a 19" rack panel and the input power connections are already wired together. The power supplies are individual 19" rack mount units. The computer has all necessary software preloaded but a backup CD is provided.

Assembly

Assembly of SVT Associates' Model VC45-As Arsenic Valved Cracker is fairly straightforward. The major sections were shipped preassembled. Only high-level assembly is necessary and can be performed by anyone with experience working with vacuum equipment. The entire source has been cleaned and outgassed prior to shipment. It has **not** been contaminated with Arsenic. Be sure to perform assembly under clean room or clean bench conditions. Observe standard practices for dealing with items to be placed in a vacuum system.

Follow these steps to assemble the VC45-As:

1. **Check resistances of heater filaments and thermocouples.** Make sure the filament and thermocouple wires are not open by doing a resistance check with a voltmeter or ohmmeter. Each filament should measure approximately 1 Ohm when at room temperature. Check the Component Data Sheets for the exact values for this source. All filaments should be isolated from the outer shell of the source. Make sure no wires are shorted to the outer case of the source. The thermocouple is isolated from ground. There should be no connection between the thermocouple and filament.
2. **Insert the baffle into the cracking tube.** Find the "T" shaped piece of metal and insert it into the long cracking tube until the top of the "T" rests on the end of the tube. Do not straighten it out. It's supposed to be twisted to disrupt the gas flow.
3. **Mount the cracking head assembly on the system.**

NOTE: This step is optional at this point but highly recommended. Assembly of the cracker can be finished on the bench and then the entire source can be mounted on the system. However, the source is rather large and heavy and depending on your system configuration, it might be easier to mount the partially assembled source at this time and then finish cracker assembly on the system. Otherwise the source can be mounted after steps 5, 6 or 7.

Using the provided hardware, place a standard 4.5" gold-plated copper gasket into the flange and bolt the source into place.

4. **Fill the moly crucible with Arsenic.**



WARNING: OBSERVE STANDARD SAFETY PROCEDURES FOR DEALING WITH ARSENIC. See MSDS for more information.

This should be done carefully, observing all precautions for dealing with Arsenic. The crucible can be filled as close to the lip as is practical depending on the mounting angle of the source and the form of Arsenic being used as long as the valve isn't blocked. A shaped charge will allow full use of the crucible's volume. The crucible cannot be filled as completely if pellets, rods, shot or ingots are used. One convenient form of Arsenic is a rod (1/4" diameter). Several of these should fit in the crucible.

NOTE: The source was outgassed before shipping. However, you may want to outgas the entire source before using it the first time. If so, do not fill the crucible with Arsenic at this time. It is safe to run the source with an empty crucible.

5. **Mount the moly crucible to the valve body.** See Figure 2. Place the crucible gasket over the flared end of the crucible. Use the three 6-32 x 0.5" titanium socket head cap screws to mount the crucible onto the

valve body in the cracking head. These fit in the untapped holes on the crucible lip. Make sure the tapered lip of the crucible seats evenly in the valve body.

6. **Mount the lower water jacket and bulk evaporator.** Place a 4.62" gold-plated copper gasket into the flange. Use the 12-point bolts which were shipped with the cracking head to secure the lower water jacket to the cracking head.
7. **Connect the water cooling tubes.** Find the separate pieces of 1/4" poly tubing. Connect the tubing as shown in Figure 6. Connect the water supply to the water inlet at the bottom of the source. Connect the water return to the 1/4" tube located at the top rear side of the source. All water jackets have NPT to Swagelok fittings supplied. The only hardware required is the supply and return line. Use 1/4" poly tubing for connection.

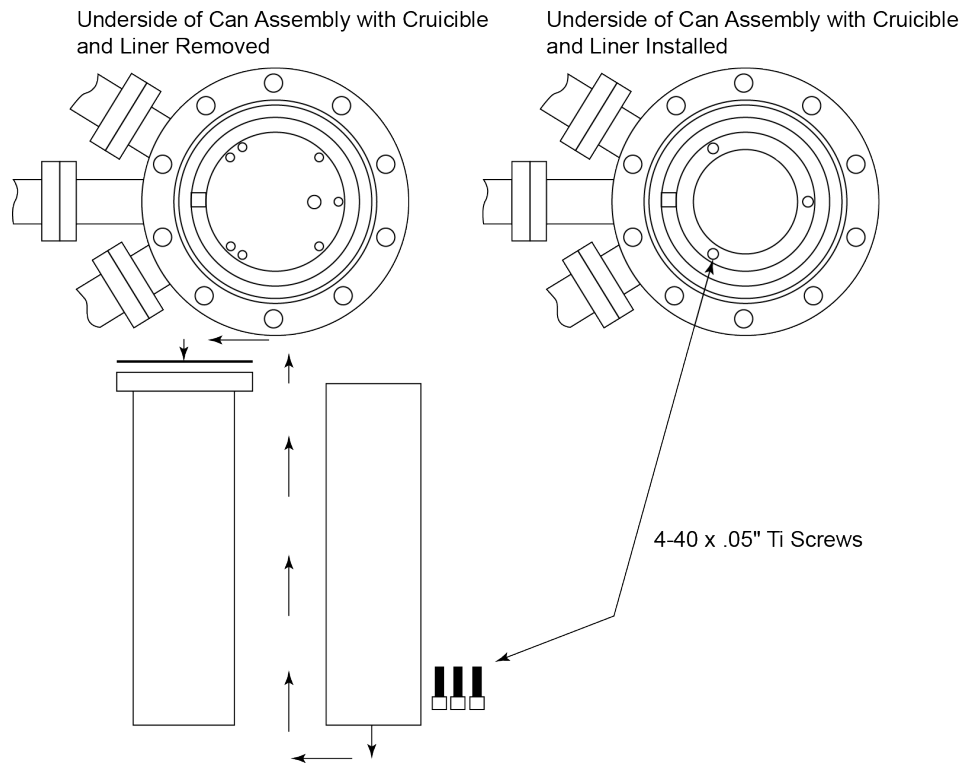


Figure 5: Mounting of the Crucible (3 or 4 screw hole pattern possible)

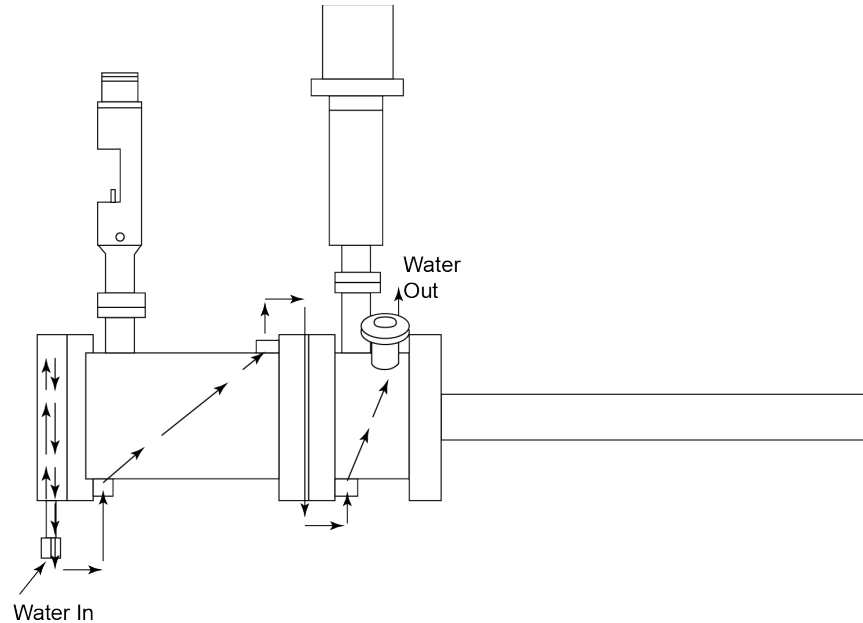


Figure6: Cooling Water Supply

Installing the Cracker into the Chamber

The VC45-As is designed to be mounted on a standard 4.5" conflat flange. If you have not already done so, insert the source into the system flange using the proper size gold-plated copper gasket and bolt into place. Make sure the water connections are oriented as shown in Figure 6. No other alignment is required.

Installing the Electronics

Mount any electronic equipment (power supplies, temperature controllers and/or motor controller) in a 19" rack within 25 feet of the cracker. Make sure the rack has adequate ventilation and power. Plug all equipment into the appropriate outlets. If a computer is being used, place it in a convenient position near the system.

Connecting the Cables

NOTE: Before connecting the cables, repeat Assembly Step 1 and check the heater and thermocouple wires for opens or shorts.

Heater/thermocouple cables: Inspect the cables and identify the two with end connectors that match those on the connector blocks mounted on the cracker. They are identical. Attach the proper connectors, heater and thermocouple, to the connector blocks on the cracker. Attach one to the tip connector block near the flange and one to the bulk evaporator connector block at the bottom of the cracker. Make sure to note which cable is which at the other ends. Attach the screw terminal wires on the other end to the proper terminals on the power supplies. There is no polarity to consider. Connect the red thermocouple connectors to JTC1 and JTC2 on the controller. Connect the two short cables with circular connectors to J11 and J12 on the controller. Connect the DB-25 ends to the two power supplies. Do not force or over tighten any connection. When properly aligned they should all go together easily.

Motor cable: If you have ordered the motor option, connect the motor cable between the connector on the linear motion feedthrough on the cracker and J14 on the rack mounted controller box.

Computer cable: If you ordered the computer option, connect the cable with the DB9 connectors between the RS485 serial port on your computer and J24 on the rack mounted controller box.

Operation

Starting the Source

Users may already have or will develop their own preferred methods of operating the source. However, use the following steps as typical operating procedure:

1. With the valve just slightly open, slowly ramp up all two heaters until reaching 200 °C for the bulk heater and 350 °C for the cracking tip heater. These are typical idling temperatures. The source is usually left in this state overnight.
2. Slowly ramp up the cracking tip heater at approximately 15 °C /minute to 900 °C. A good operating range is 800 °C to 1000 °C.
3. After about 20 minutes, ramp up the bulk (Arsenic crucible) heater slowly at approximately 10 °C /minute to 450 °C. A good operating range is 400 °C to 600 °C.
4. Slowly open the Valve until the desired flux rate is achieved. This will have to be determined experimentally. If you have the Motor Control Option, operate the valve using the switch on the Controller or from the computer if that option has been purchased and installed.

Turning Off the Source

- Close the valve.
- Slowly open the Valve until the desired flux rate is achieved. This will have to be determined experimentally. If you have the Motor Control Option, operate the valve using the switch on the Controller or from the computer if that option has been purchased and installed.

Turning Off the Source

- Close the valve.
- Ramp down the bulk (Arsenic crucible) heater slowly to 200 °C.
- Ramp down the Cracking Head Tip Heater slowly at approximately 15 °C /minute to 350 °C.
- The cracker is now in its idle state and may be left like this overnight.

Note: Always use power limits when operating any zone of your As source. This avoids potential damage to filaments. Settings are specified in your source component sheets.



Warning: NEVER move the valve pin when the valve body is cold. Potential condensation of As can harm the coupler or pin mechanics. Always move the valve in hot condition (that means bulk and cracking heater on).

As Cracker Outgassing

Outgassing w/o material (empty): With liquid nitrogen through shroud and pressure < 10⁻⁶ torr and valve fully open:

As bulk zone	As cracking head
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Ramp to 500 °C at 5 °C Per Minute	Ramp to 1100 °C at 7 °C Per Minute
Hold at 500 °C for 1-3 Hours	Hold at 1100 °C for 1-3 Hours
Ramp to 150 °C at 5 °C Per Minute	Ramp to 300 °C at 10 °C Per Minute

Note: The As valve body is parasitically heated by the As bulk zone and does not typically require separate active heating.

Outgassing with material loaded: With liquid nitrogen through shroud and pressure < 10^{-6} torr and As shutter and valve fully open:

As bulk zone **	As cracking head
Ramp to 350 °C ** at 5 °C Per Minute	Ramp to 1100 °C at 7 °C/min
Hold at 350 °C for 1 Hour	Hold at 1100 °C for 1 Hour
Ramp to 150 °C at 5 °C Per Minute	Ramp to 300 °C at 10 °C Per Minute

** Normal As bulk temperatures used during growth are in the 300 °C – 400 °C range. Monitor your system pressure and mass spectrometer (RGA) signal. If too much Arsenic flux is present during this outgassing step, outgassing time and/or As, bulk temperature should be reduced.

Initial Calibration of Arsenic Valved Cracker Source

These instructions assume the cracker source has been filled with Arsenic and the growth system has been baked and is ready for use. Idle temperatures of the source while under vacuum:

Cracker: 300 °C

Bulk region: 150 °C

1. Check/establish cooling water flow through the Arsenic source. This cooling water prevents the outer housing from becoming too warm during use. Water flow is on the order of 0.1 – 0.5 gallons per minute (0.4 – 2 liters per minute). Flow only the minimum amount necessary to keep the outer jacket of the As cracker housing < 50 °C.
2. Establish liquid nitrogen flow through your MBE system. Proceed to step 3 when sufficient liquid nitrogen exists in the source shroud.
3. Ramp As cracker head from 300 °C to 1000 °C at 10 °C per minute.
4. Ramp As bulk from 150 °C to 270 °C at 5 °C per minute. **Keep the As valve fully open.** Also open the As source shutter. This step may be started at the same time as step #3.
5. If an RGA (mass spectrometer) is available, monitor the As signal at ~ 74 amu. Check for an increase in the As peak during the As source warm up.
6. If an RGA or flux monitor is in a good position to measure flux from the As source, start ramping the As bulk region to 350 °C at 5 °C per minute. Monitor the RGA/flux signal during this ramp.
7. When sufficient As-flux is being supplied by the As-source, immediate changes in pressure can be observed when the As shutter is opened/closed, or the valve position is adjusted. Typical ambient growth chamber pressures (as measured by an ion gauge) for As-based growths are in 1×10^{-8} to 5×10^{-7} torr range. Pressures higher than 1×10^{-6} torr could indicate too much Arsenic flow.

If As flux is still too low, increase the As bulk region temperature, not to exceed 450 °C. Operate the Arsenic bulk at the near-minimal temperature required to provide sufficient flux with the valve well open.

Running at operating temperature with valve fully closed for extended periods of time is not recommended.

Suggested method for initial As calibration:

Load an epi-ready GaAs wafer into the growth system and desorb the native oxide layer (desorption typically occurs at 580 °C). A line pattern on the RHEED system should verify a clean GaAs surface. Grow GaAs on the GaAs substrate at a temperature ~ 550 °C. If the RHEED pattern disappears in a short period of time (< 2 minutes), the As flux may be too low.

Depending on the thickness, a Ga-rich surface will appear to be hazy/silvery upon visual inspection. A proper GaAs growth will maintain a shiny black appearance. RHEED oscillations can also provide real-time feedback about growth conditions.

Ramp Down of Arsenic Valved Cracker Source

1. After using the As source for growth, ramp the As bulk region to 150 °C at 5 °C per minute.
2. When the As bulk region is < 250 °C, start ramping the cracker. As cracker -> 300 °C at 10 °C per minute.

Ramp Up of Arsenic Valved Cracker Source for Growth

1. Establish liquid nitrogen flow through your MBE system. Proceed with step 2 when sufficient liquid nitrogen exists in the source shroud.
2. Ramp As cracker head from 300 °C to 1000 °C at 10 °C per minute.
3. Ramp the As bulk region to the temperature established for growth conditions at 5 °C per minute (typically in 300 °C – 450 °C range). Make sure the valve is in the open position.

Maintenance

The VC45-As is relatively maintenance free. However it will be necessary to fill the crucible occasionally. This is a simple procedure, which can be performed by any competent operator.



WARNING: OBSERVE STANDARD SAFETY PROCEDURES FOR DEALING WITH ARSENIC.

Procedure:

1. Remove the water connections.
2. Remove the entire cracker from the system if desired.
3. Remove the bulk evaporator lower water jacket.
4. Remove the three 6-32 x 0.5" titanium socket head cap screws. The crucible will probably be stuck tight to the valve body at this point. Slowly turn it until the crucible breaks free. Remove the crucible and refill.
5. Replace the crucible and reassemble the source. It is suggested that the three 6-32 x 0.5" titanium socket head cap screws be replaced periodically as they have a tendency to eventually seize up and possibly break off. They will then have to be drilled out.

Appendix

Specifications

Valved Cracker: – Model VC45-As-200 (Arsenic)

Mounting Flange	4.50" CF
Length (inside flange to end of cell)	12.0"
Diameter	1.4"
Capacity	200 cc
Max. Bulk Evaporator Temperature/Power	500 °C/150 Watts
Max. Cracking Head (Tip) Temperature/Power.....	1200 °C/200 Watts
Source Temperature Stability	± 0.1 °C
Source Temperature Reproducibility	± 0.1 °C
Thermocouple Type	Type C
Electrical Connectors	Filament: Amphenol Circular T/Cs: Omega Subminiature

Options

- Two Power Supplies
- Two Temperature Controllers
- Valve Motor and Controller
- Computer Interface
- Computer
- Water Shroud for Cracking Head

MSDS Arsenic



General

Synonyms:	arsenic black, arsenicals, arsenic-75, colloidal arsenic, grey arsenic, metallic arsenic
Molecular formula:	As
CAS No:	7440-38-2
EINECS No:	231-148-6
Annex I Index No:	033-001-00-X

Physical data

Appearance:	grey powder
Melting point:	817 C
Density (g cm ⁻³):	5.727

Stability

Stable. Incompatible with acids, oxidizing agents, halogens. Heat and air-sensitive.

Toxicology

Very toxic. May be fatal if inhaled, swallowed or absorbed through the skin. This is a known human carcinogen. May cause reproductive disorders.

Toxicity data

- ORL-RAT LD50 763 mg kg⁻¹
- IPR-RAT LD50 13 mg kg⁻¹
- ORL-MUS LD50 145 mg kg⁻¹
- IPR-MUS LD50 46 mg kg⁻¹
- Risk phrases
R26 R27 R28 R45 R47.

Transport information

CGD UK Major hazard class: 6.1. Packing group: II

Personal protection

Gloves, safety glasses, good ventilation. Handle as a carcinogen.
Safety phrases
S20 S21 S28 S45.

Warranty and Limitations of Remedies

SVT Associates warrants that all equipment manufactured by it shall be free from defects in materials and workmanship under normal use and service for a period of twelve (12) months from the date of shipment from SVT Associates manufacturing facility. This warranty is subject to SVT Associates equipment being installed, maintained, and operated in accordance with the operating and maintenance instructions accompanying each item manufactured by SVT Associates. Warranty shall be void if SVT Associates equipment is modified by the CUSTOMER or used in other than the recommended manner or applications. Purchased equipment incorporated into any item supplied by SVT Associates will be covered by said manufacturer's warranty.

SVT Associates warrants that, at the time of delivery, any other products processed or manufactured and sold by it hereunder are free of defects in material and workmanship and conform to COMPANY specifications. No warranty is provided by SVT Associates for products sold hereunder which are not manufactured or processed by SVT Associates, but the manufacturer's warranty for such products, if any, shall be assigned to the CUSTOMER without recourse to SVT Associates. The foregoing warranties are in lieu of and exclude all other warranties not expressly set forth herein, whether expressed or implied by law or otherwise, including without limitation any warranty of merchantability or fitness for a particular purpose. In no event will SVT Associates be liable for any consequential damages.

IN THE EVENT OF SVT ASSOCIATES LIABILITY, WHETHER BASED ON CONTRACT, TORT (INCLUDING BUT NOT LIMITED TO NEGLIGENCE AND STRICT LIABILITY) OR OTHERWISE, THE CUSTOMER'S SOLE AND EXCLUSIVE REMEDY WILL BE LIMITED; SVT ASSOCIATES HAS THE FOLLOWING OPTIONS; TO REPAIR OR REPLACEMENT (F.O.B. SVT ASSOCIATES MANUFACTURING PLANT) BY THE COMPANY OF ANY NON-CONFORMING ITEM FOR WHICH CLAIM IS MADE BY THE CUSTOMER OR TO REPAYMENT OF THE PORTION OF THE PURCHASE PRICE PAID BY THE CUSTOMER ATTRIBUTABLE TO THE NON-CONFORMING ITEM. SVT ASSOCIATES WILL NOT BE LIABLE FOR ANY OTHER DAMAGES, WHETHER DIRECT, INCIDENTAL, CONSEQUENTIAL OR OTHERWISE.

Return Policy

Any request by the CUSTOMER for return of standard products other than for warranty claims under warranty hereof, for all or any part of purchase order accepted by SVT Associates, shall be subject to the following conditions:

- A. The CUSTOMER must make notification to SVT Associates within thirty (30) days of original shipping date.
- B. A "RETURN GOODS AUTHORIZATION" number must be assigned to and accompany all goods or materials being returned by the CUSTOMER to SVT Associates. SVT Associates must assign said number prior to any and all returns. Goods not accompanied by a "RETURN GOODS AUTHORIZATION" number will be refused by SVT Associates and returned at the CUSTOMER'S expense.
- C. CUSTOMER shall prepay shipping charges for products being returned to SVT Associates.
- D. Products being returned to SVT Associates should be properly crated for shipment, and the CUSTOMER shall bear the risk of loss until delivered to SVT Associates.
- E. Products being returned to SVT Associates must be returned in the condition originally received by the CUSTOMER and free from damage, use, or modification, which would render the product unusable for resale, by SVT Associates.
All applicable taxes, duties, insurance, and shipping charges shall be the sole responsibility of the CUSTOMER.
- F. Goods being returned for other than warranty repair shall be subject to a restocking charge of twenty (20) percent of the original sales price of the returned item.



Returning Equipment for Repair or Servicing

Before shipping equipment for repair or servicing, obtain a Return Authorization Number assigned by SVT Associates.

Liability Disclaimer

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