

# K87170 AUTOMATIC VACUUM DISTILLATION SYSTEM

# **OPERATION AND INSTRUCTION MANUAL**

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## Contents

General Information1-1
Description of Equipment
System Preparation
Operating the Controller
Performing a Distillation5-1
Maintenance and Trouble Shooting6-1
Replacement Parts7-1
Glossary

## **Requirements for Installation of Automatic D1160 System**

### **Electrical Requirements.**

Two single phase 120 or 220/240 VAC., 50 Hz/60 Hz, dedicated electrical outlets within 3 feet of the installation site. The amperage required depends on the particular system ordered. Please specify Voltage and Hz when ordering. The electrical power must be clean. Fluctuations in the power or noise on the power line may cause equipment malfunctions. Any abnormalities in power should be reported in detail at the time of ordering.

### Nitrogen

As an option, gaseous nitrogen can be used as a bleed gas for vacuum control instead of air. If nitrogen is to be used as the bleed gas, a gaseous nitrogen source is required. The cylinder should be equipped with a two stage regulator with a hose barb adapter to accommodate a 3/8" ID hose. *The regulator should measure in 1 lb. increments, up to 30 lbs.* 

### **Manual Cold Trap**

The manual cold trap requires dry ice and acetone.

### Venting.

A vent to the outside should be provided to vent the vacuum pump exhaust.

### Environment

The area in which the instrument is operated should be free from all ignition sources such as sparks and flames and be well ventilated. Flammable solvents should not be stored within 5 feet of the system unless those solvents are contained in a flammable storage cabinet.

The area should also be temperature controlled between 20°C (68 °F) and 27°C(81°F) with a relative humidity less than 80%.

### Cleaning

A source of reagent toluene or cyclohexane should be available for cleaning the unit after each run.

### Freight

This equipment is very fragile. There are many delicate glass pieces and electronics packed with the unit. Koehler takes great care and expense in packing this equipment. The customer should take all possible precautions to ensure that the equipment is handled carefully and given a smooth ride during shipment. Keep in mind that this is a very delicate and valuable piece of equipment. Please see to it that it is treated as such.

Please remember that the technician will be traveling a long distance to install and train laboratory personnel. Having the site properly prepared will lead to quick installation and allow more valuable time to test the unit and train lab operators! Please pay careful attention to each of the requirements listed above. If you have any questions please contact Koehler Instrument for advice. Thank you for your attention to this matter.

## **Reporting Damaged Equipment**

**Damage due to shipment must be reported immediately to both the carrier and Koehler Instrument Company, Inc.** Neither the carrier nor Koehler will be responsible for damages reported after 14 days from shipment. It is the customer's responsibility to report any damage to the carrier. Please call 1 631 589 3800 to report damage to Koehler Instrument.

### Cautions

Read all instructions carefully before unpacking your system.

Installation and training is provided by a trained technician. All operators should be trained in the safe and proper operation of the system. Training is provided during installation, but you may feel that more training is necessary at a later date. To arrange for further training, please contact us.

Your company should develop standard operating procedures for safe and proper operation of the D1160 System.

For your protection, never plug in and turn on the power until the installation has been completed in its entirety.

Laboratories using flammable solvents should be protected with an automatic fire extinguishing system.

Goggles, gloves, and other protective safety equipment should be provided and used when handling petroleum products and chemicals.

### **Description Fully Automatic D1160 Components**

#### Figure 1

<u>Vapor Temperature Probe</u>: This temperature probe measures the actual vapor temperature.

<u>Pot Temperature Probe</u>: This temperature probe measures the temperature of the liquid in the pot flask.

Controller: Controlls the distillation system and includes display and keyboard.

IBP Sensor: Automatically detects first drop (Initial Boiling Point).

<u>Receiver and Level Follower</u>: This is where the distillate is collected and the volume is measured automatically.

Heating Mantle: Provides heat to the pot flask.

Vacuum Sensor: Senses the vacuum level.

<u>Vacuum Bleed Valve</u>: This automatically operated valve allows air or nitrogen into the distillation system to regulate the vacuum level.

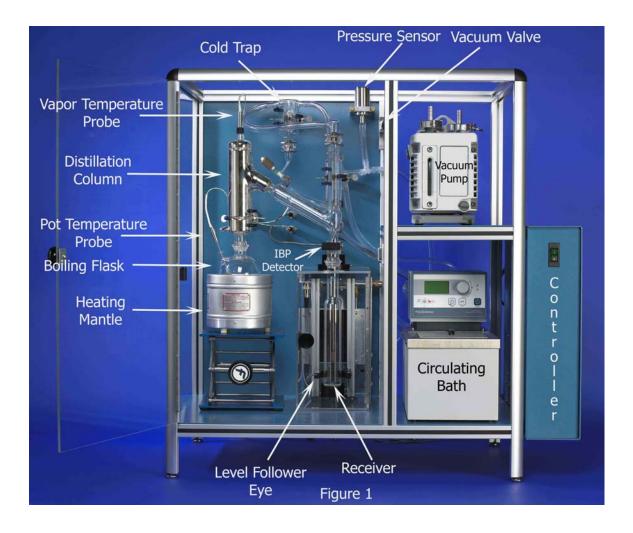
<u>Cold Trap</u>: Filled with dry ice (solid CO2). The cold trap protects the vacuum pump and pressure sensor from uncondensed gases.

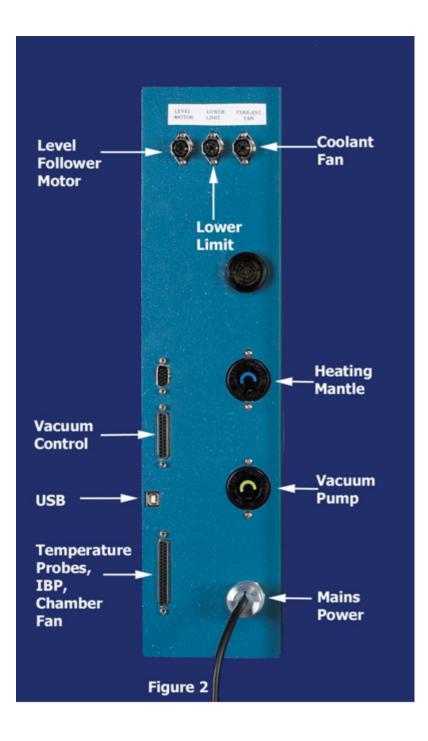
Distillation Column: Column for distilling the sample.

Pot Flask: Flask for distilling the sample.

#### Figure 2

Electrical connections on back of M690 controller.





### **Preparing the System**

#### **Operational Checks**

- 1. Check the condenser bath level. The level should be maintained above the coil inside the bath.
- 2. Verify that a clean receiver is in place and empty. The metal drip trough should be pointing directly to the front of the unit. The metal drip trough should also be touching the side of the receiver.
- 3. Verify that the receiver joint is greased.
- 4. Put dry ice in the cold trap.
- 5. Check the vacuum pump oil level. It should be near the high mark.

#### Electrical System Check

- 1. The Control Unit power should be in the ON position.
- 2. The Condenser Bath power switch should be in the ON position.
- 3. The Vacuum Pump power switch should be in the ON position.

## General

The D1160 control software is designed to make vacuum distillation as easy as possible.

The Main Screen is displayed when the program is started. It gives various options for common tasks

ar M690 PC Int	lerface				
File Help					
1.1					
	Manual Controls	Manage Run Data			
	Manual Controls	Manage nun Data			
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	View / Print Results	Start Vacuum Distillati	on		
	System Options	Start Cleaning Run			
	0				
	Calibration				
🛃 start	M690_Results	🖙 Removable Disk (E:)	関 run data graph2.rtf	Jar M690 PC Interface	🧟 😵 11:52 AM

**Manual Controls:** This area is used for manually operating all aspects of the equipment. The vacuum pump, bath, level follower and so forth can be manually operated. At the same time vacuum level and various temperatures can be monitored. This is useful for checking the equipment function and performing maintenance.

Manage Run Data: This area is used to create and edit distillation programs.

**View and Print Results:** This area allows data from distillations to be viewed or printed.

Operating the Microprocessor Controller 4-1

Start Vacuum Distillation: This area is used to start a vacuum distillation run.

**System Options:** This area is used to turn on/off the various options available with this equipment

Start Cleaning Run: This area is used to start a cleaning cycle.

**Calibration:** This area is used to calibrate the pressure, temperature and volume measurements.

## **Manual Controls**

The Manual Controls button launches the Manual Control screen shown below.

🕼 M690 Manual Controls			_ 2 2
Image: style styl	Discreet Inputs Coolant Flow Spare In 7 Spare In 8 Spare In 9 Spare In 1 Spare In 2 Spare In 3 Spare In 4 Spare In 5 Spare In 6 Keep Alive Test Heating Mantle Power Rate 10.0 %	Discreet Outputs Band Motor Reflux Valve Spare Out 7 Coolant Valve Spare Out 1 Spare Out 2 Spare Out 3 Spare Out 3 Spare Out 4 Spare Out 5 Spare Out 6  Level Follower Find Upper Limit Counts 0 Up Slow Sensor Clear Track Down Slow Outme 0.0 mL Down Slow Find Base Lower Limit Off Standby	
Removable Disk (E:)	🚽 M690 Manual Controls		🕜 🗞 🛞 9:50 AM

**Transducers** show the current temperatures for the pot temperature probe and the vapor temperature probe.

**Vacuum Control** area is used for testing the vacuum system. Once the pump is turned on enter the desired vacuum level in the Set Point field. The current pressure can be viewed in the Pressure field.

The **Condenser Bath** can be turned on by checking the box next to the word Power. Enter the desired temperature in the Set Point field. The current temperature is displayed in the Temperature field. The Serial Data field shows the current serial communications between the computer and the bath.

Drop Count displays the number of drops the have been detected by the IBP detector.

The **Heating Mantle** heaters can be turned on by checking the box next to the word Power. Enter the percentage of the total Wattage desired in the Heat Percent field.

The Level Follower has a number of manual control options. Find Upper Limit sends the level follow to the top of its range. Up Slow sends the level follower up slowly. Track mode instructs the level follower to find the current liquid volume in the receiver and follow it if it increases. Down Slow sends the level follower down slowly. Find Base sends the level follower down till the base is found. Standby cancels previous commands and leaves the level follower in the rest position.

There are also a variety of outputs from the level follower. **Counts** tells how many steps the level follower is from the base. **Sensor** indicates if the level follower eye is **blocked** (liquid is blocking the light from the emitter to the detector). **Clear** indicates liquid is not blocking the light from the emitter to the detector. **Volume** tells the volume of liquid that corresponds to the current level follower position. **Lower Limit** indicates if the lower limit detector is blocked (on) or not blocked (off).

## Manage Run Data

To edit or create a distillation program select LOAD from the File menu.

ler M690 D	1160 Run Data			
File Help				
🗃 Load				
Save				
Exit				
	Pressure Set Point:	10.00	mmHg Show Tuning Parameters	
	Initial Heat:	25.0	%	
	Initial Take Off Heat:	30.0	%	
	Target Distillation Rate	7.0	mL/Min	
	Max Pot Temp:	400.0	°C	
	Condenser Temp:	60.0	°C .	
	Pressure Step Down Rate:	50.0	mm Hg / min	
	Max Vapor AET:		r	
	Distillation Rate PID Bias	25.0		
	Stop by Volume			
	Stop on Cracking	Detection		

Select the desired program from the list.

M690 D1160 Run Data le Hep		. 2)
Pressure Set Point:     10.00     mmHg       Initial Heat:     25.0     2       Initial Take 0ff Heat:     30.0     2       Target Distillation Rate     7.0     mL/Mn       Max Pot Temp:     400.0     °C       Condenser Temp:     60.0     °C       Pressure Step Down Rate:     50.0     mm Hg / min       Max Vapor AET:     °C       Distillation Rate PID Bias     25.0       Stop by Volume     Stop on Cracking Detection	Open D11 60 Run Data         Look in:       M 590_Runs <ul> <li>Image: Colspan="2"&gt;Image: Colspan="2"&gt;Image: Colspan="2" Image: Colspa="2" Image: Colspan="2" Image: Colspan="2" Image: Colspa</li></ul>	Open       Cancel

Edit the parameters as desired and then save the distillation program with the desired program name.

	1160 Run Data						
File Help							
	(*						
	Pressure Set Point:	10.00	mmHg	Show Tuning Parameters			
	Initial Heat:	25.0	*				
	Initial Take Off Heat:	30.0	%				
	Target Distillation Rate		mL/Min				
	Max Pot Temp:	400.0	°C				
	Condenser Temp:	60.0	°C				
	Pressure Step Down Rate:	50.0	mm Hg / min				
	Max Vapor AET:		°C				
	Distillation Rate PID Bias	25.0					
	Stop by Volume						
	🔲 Stop on Cracking I	Detection					

- a. **Pressure Set Point**: the range is 1 to 50 mmHg. The default is 10 mmHg.
- b. **Initial Heat Rate**: This is the amount of heat that will be applied to the pot flask until the first drop is detected. The value is a percentage of the total available wattage for the heating mantle. The default value is 50%. If the distillation rate is too high during the beginning of the distillation then lower this value. If the distillation rate is too low during the beginning of the distillation then raise this value.
- c. **Initial Take Off Heat Rate**: This is the amount of heat that will be applied to the pot flask from the the time that first drop is detected till 10% of the volume is distilled. If the distillation rate is too high during the this time then lower this value. If the distillation rate is too low during this time then raise this value.
- d. **Target Distillation Rate:** This is the desired distillation rate in ml/min. ASTM D1160 specifies an averaged distillation rage of 6-8 mL/min.
- e. **Maximum Pot Temperature**: This is the maximum pot temperature allowed for this distillation. The default value is 300 °C. The maximum allowable value is 350 °C for borosilicate flasks and 400 °C for quartz flasks. If the pot temperature exceeds 300 °C for a borosilicate flask during a distillation it should be checked for strain before being returned to service.

- f. **Condenser Temperature**: Set the temperature of the condenser coolant to at least 30 °C below the lowest vapor temperature to be observed in the test. A suitable coolant temperature for most distillations is 60 °C.
- g. **Pressure Step Down Rate:** This the rate at which pressure will be reduced before the distillation begins. Pressure step down begins at 100 mmHg and is reduced from there at the desired rate. Slow reduction in pressure helps to prevent foaming by allowing the sample to slowly degas before the distillation begins.
- h. **Maximum Vapor AET**: This is an *optional* field in case there is a need to stop the distillation at a specific vapor temperature. If the field is left blank, it appears grey and is ignored. If the field has a value entered then, the distillation will stop at that atmospheric equivilant temperature (AET).
- i. **Distillation PID Bias:** This is the starting point for the distillation rate control. If the distillation rate is too high then lower this value. If the distillation rate is too low during then raise this value. Normally the default value is suitable.
- j. **Stop by volume**: If this box is checked, then an additional field appears. This is an *optional* field in case there is a need to stop the distillation at a specific volume % distilled. If the box is not checked then the field is ignored. If the box is checked the the distillation will stop at the volueme % entered in the field.
- k. **Stop on cracking detection**: This is an optional function. When check, the system will monitor the system for signs of cracking of the sample. If sample cracking is observed, the distillation will stop automatically. If the box is left unchecked, then this function is disabled.

"Hidden" Parameters

There are some parameters that rarely need adjustment. These are normally "hidden" from view to avoid clutter and confusion. To view or edit the parameters, click the Show Tunig Parameters button. Then these parameters will be visible.

) D1160 Run Data					
Pressure Set Point:	10.00	mmHg	Hide Tuning Parameters	Distillation Rate Control	
Initial Heat:	25.0	%	Above 10 Vacuum KP: 40	Distillation Rate KP: 7.0	
Initial Take Off Heat:	30.0	%	Above 10 Vacuum KI: 200	Distillation Rate KI: 9.0	
Target Distillation Rate	7.0	mL/Min	1 to 10 Vacuum KP: 6	Distillation Rate KD: 5.0	
Max Pot Temp:	400.0	°C	1 to 10 Vacuum KI: 30		
Condenser Temp:	60.0	°C	Below 1 Vacuum KP: 2		
Pressure Step Down Rate:	50.0	mm Hg / min	Below 1 Vacuum KI: 10		
Max Vapor AET:		°C			
Distillation Rate PID Bias	25.0				
🔽 Stop by Volume	100.0	%			
🔲 Stop on Cracking	Detection				

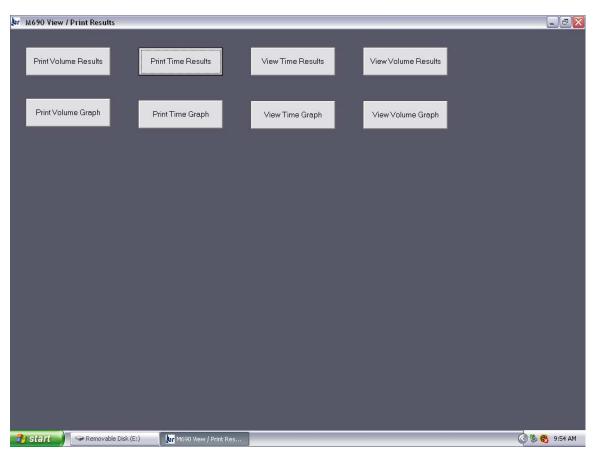
There are 3 sets of PID control parameters for the automatic vacuum control. The first set is for pressures above 10 mmHg, the second for 1-10 mmHg and the third for pressures below 1 mmHg.

- a. **Vacuum KP:** This is the proportional control constant for the vacuum control. The default value is normally adequate and should not need to be changed. If you are having problems with vacuum control contact your local representative for advice.
- b. **Vacuum KI:** This is the integral control constant for the vacuum control. The default value is normally adequate and should not need to be changed. If you are having problems with vacuum control contact your local representative for advice.
- c. **Distillation Rate KP:** This is the proportional control constant for the distillation rate control. Normally this value does not need to be changed. If you are having problems with distillation rate control contact your local representative for advice.
- d. **Distillation Rate KI**: This is the integral control constant for the distillation rate control. Normally this value does not need to be changed. If you are having problems with distillation rate control contact your local representative for advice.
- e. **Distillation Rate KD:** This is the differential control constant for the distillation rate control. Normally this value does not need to be

Operating the Microprocessor Controller 4-8

## **Print and View Results**

The primary Print and View Results Screen is below.



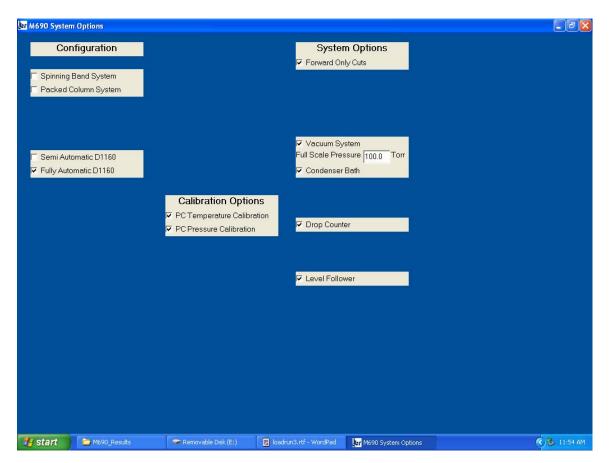
The **Print Volume Graph** button allows a graph of Temperature versus Volume to be printed.

The **View Volume Results** button allows the temperature versus volume data to be viewed.

The **Print Volume Graph** button allows a graph of Temperature versus Volume to be printed.

The **View Volume Results** button allows the temperature versus volume data to be viewed.

## **System Options**



System Options for the fully automatic D1160 should be set as seen below.

## Calibration

The main calibration screen has options for calibrating temperature, pressure and volume.

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			NO	
		Temperature (	Calibration	
		1		
		Pressure Ca	libration	
		Volume Cal	Ibration	
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## **Temperature Calibration**

nperature Calibration			
Vapor Probe			
	0	Raw Data	
Low Calibration Point 24.0	°C 19061	Raw Data	
High Calibration Point 150.4	°C 27417	Raw Data	
130.4			
Pot Probe			
	-		
	0	Raw Data	
Low Calibration Point 24.0	°C 18996	Raw Data	
Low Calibration Point 124.0			
High Calibration Point 150.4	°C 27233	Raw Data	

## **Pressure Calibration**

Pressure Calit e	bration					
		100 mm H	lg Pressure S	ensor		
Calibration Point	Sensor 0	utput	Calibration	Data		
C Point 1		Volts	0	Counts	1.1	mm Hg
C Point 2		Volts	498	Counts	12.3	mm Hg
C Point 3	-	Volts	1254	Counts	30.6	mm Hg
C Point 4		Volts	2504	Counts	61.1	mm Hg
Discreets			Parameters			
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🔽 Vacuum Pu	umo 1	0	КІ			
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start	Semo'	vable Disk (E:)	) Jur	Pressure Ca	ibration	

## **Volume Calibration**

er M690 Volume Calibration			
File			a construction of the second states and the second states and the second states and the second states and the second states are second states and the second states are second stat
File  Level Follower Controls  Find Base  Track  Standby  Actual Counts  0	Counts		
<b>H start</b> M690 Volume Calibrat	💝 Removable Disk (F:)	Document.rtf - Word	🔦 🗞 8:57 AM

Calibration of the receiver volume is performed from the screen shown above.

- 1. Put the empty receiver in place and select **Find Base**.
- 2. After the level follower finds the base, select **Stand By**.
- 3. Remove the receiver and use a class A pipette to deliver a known volume of toluene into the receiver.
- 4. Put the receiver in place and select **Track**.
- 5. The level follower should move up to the level of the toluene in the receiver.
- 6. Enter the volume of toluene that was pipetted into the receiver in the Calibration Volume Field.
- 7. When the level follower has stopped moving click on the **Get Counts** button.

- 8. Click on the **File** menu in the upper left hand corner of the screen and select **Save File**.
- 9. Enter the file name of VCal.dat and click on **Save.**

ur M690 Volume Calibration						<b>.</b> 8 X
File						
Level Follower Controls	Save Volume Ca				28	
F Find Base F Track F Standby Actual Counts 0	Save in: My Recent Documents Desktop My Documents	№ М690			<b>ĕ</b> ∎•	
Calibration Counts 2923 Get Counts Calibration Volume 100.0 mL	Places	File name: Save as type:	VCal.dat Cal Data Files(VCal.dat)		▼ Save ▼ Cancel	
🛃 start 🛛 🐙 M690 Volume Calibrat 🚺 Save	Volume Calibrati	🗢 Removable Dis	k (F:) 🛛 🙀 Docume	nt2.rtf - Wor		🔦 😼 8:58 AM

- 10. Now the calibration data is saved. You can check the calibration in Manual Controls.
  - a) Put the empty receiver in place
  - b) Select Find Base.
  - c) Remove receiver and pipette into the receiver a known amount of toluene
  - d) Put receiver in place and select Track
  - e) Record the displayed volume when the level follower reaches the top of the liquid and stops moving.

- 1. From the density of the sample determine the weight, to the nearest 0.1 gram, equivalent to 200 ml of the sample at the temperature of the receiver. Weigh this quantity of oil into the distillation flask. Add boiling chips.
- 2. Make sure the anti-splash guard is in the neck of the receiver. The trough should be touching the receiver wall and should be pointing to the front of the unit. This ensures that the distillate will travel down the front wall of the receiver and will not drip causing splashes.
- 3. If required, make sure the stainless mesh is in the bottom of the distillation column. The mesh is used to break up any foaming of the sample.
- 4. Follow all pre-run procedures in **Section 3** of this instruction manual.

### CAUTION

Fluids, such as petroleum, crude oil, solvents, reagents, and water, should not be spilled on the surface of the mantle. Equipment failure may occur.

- 5. Lubricate the spherical joints of the distillation apparatus with suitable grease. Make certain that the surfaces of the joints are clean before applying the grease, and use only the minimum quantity required. Connect the flask to the lower spherical joint of the distilling head, place the heater under the flask, put the top mantle in place and connect the rest of the apparatus using clamps to secure the joints.
- 6. Place a few drops of silicone oil in the bottom of the thermowell of the flask and insert the temperature sensor to the bottom.
- 7. Double click the D1160 program icon on the computer. This launches the D1160 distillation program.
- 8. Select **Start Distillation** to begin the distillation.

9. Using the file menu choose LOAD

ler M690 I	D1160 Run Data								
File Help									
🗃 Load									
Save Save									
Exit									
	Pressure Set Point:	10.0	mmHg	Distillatio	n Rate Control				
	Initial Heat:	50.0	%	Distillation Rat	e KP: 10.0				
	Initial Take Off Heat:	50.0	%	Distillation Ra	ite KI: 1.0				
	Target Distillation Rate	0.0	mL/Min	Distillation Rat	e KD: 1.0				
	Max Pot Temp:	120.0	۰C	Distillation Rate PI	D Bias 50.0				
	Condenser Temp:	45.0	°C						
	Vacuum KP:	2							
	Vacuum KI:	10							
	Pressure Step Down Rate:	0.0	mm Hg / min						
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10. Then choose the desired distillation program from the list.

11. Once the program is selected, the distillation program will begin. You will be prompted for a folder name where the results will be stored. The default folder name is the current date and time. This may be changed as desired. The name must have letters and numbers only.

Jur M690 D1160	
Enter Sample ID for D1160 Distillation	
18Mar2006_11_55	
Accept	

12. Another prompt will appear where operator identification can be entered. Entering data in this field is optional.

Jur M690 D1160	
Enter Operator ID for D1160 Distillation	
,	
Accept	

br M690 D116	iO					
Volume IBP 0% 5% 10% 20% 30% 40% 50% 60% 70% 80% 90% 95% 80%	Vapor AET [453.8 'C [453.8 'C [500.0 'C [	Pressure Set Point: 1 Initial Heat Rate: 7/ Initial Take Off Heat: 6/ Target Distillation Rate 7 Max Pot Temp: 7/ Condenser Temp: 7/ Vacuum KP: 2 Vacuum KI: 11 Pressure Step Down Rate: 11 Run Mode: Lool Alarm: None	20         %           3.0         %           0         mL/Min           00.0         °C           0.0         °C	Distillation Rate Con Distillation Rate KP: 15 Distillation Rate KI: 20 Distillation Rate KD: 5. Distillation PID Bias: 50	0	
7		Pause				
Pot Tempera 291.1		Vapor AET 508.1 *C	Pressure 0.9mmHg		l'emperature	Receiver Distillation Volume Rate 19.1 mL 7.2 mL/Min
🛃 start	M690_Results	Removable Disk (E:)	Jur M690 D116	i0 III run proces	ss diagram	() 11:06 AM

13. Once the distillation has started the following screen will by displayed.

On the left side of the screen is a display of the results for this distillation including IBP (Initial Boiling Point), various distillation points and the FBP (Final Boiling Point).

On the right side is a list of the control parameters. These may be edited during the distillation.

Pressure Set Point is the desired vacuum level.

**Initial Heat Rate** is the amount of heat that will be applied to the pot flask until the first drop is detected. The value is a percentage of the total available Wattage for the heating mantle.

**Target Distillation Rate** is the desired distillation rate. ASTM D1160 method specifies an average rate of 6-8 mL/min.

**Maximum Pot Temperature** is the maximum pot temperature allowed for this distillation. The maximum allowable value is 350 °C for borosicate pot flasks and 400 °C for quartz flasks. If the pot is run above 300 °C it should be checked for strain before being returned to service.

**Condenser Temperature** is the temperature of the condenser and receiver. It should be set to at least 30 °C below the lowest vapor temperature to be observed in the test. A suitable condenser temperature for most distillations is 60 °C.

**Vacuum KP** is a proportional control constant for the vacuum control. If you are having problems with vacuum control contact your local representative for advice.

**Vacuum KI** is an integral control constant for the vacuum control. If you are having problems with distillation rate control contact your local representative for advice.

**Pressure Step Down Rate:** This the rate at which pressur will be reduced before the distillation begins. Pressure step down begins at 100 mmHg and is reduced from there at the desired rate. Slow reduction in pressure helps to prevent foaming by allowing the sample to slowly degas before the distillation begins.

**Distillation Rate KP** is a proportional control constant for the distillation rate control. If you are having problems with distillation rate control contact Koehler Instrument or your local representative for advice.

**Distillation Rate KI** is an integral control constant for the distillation rate control. It does not normally need to be changed. If you are having problems with distillation rate control contact your local representative for advice.

**Distillation Rate KD** is a differential control constant for the distillation rate control. If you are having problems with distillation rate control contact or your local representative for advice.

**Distillation PID Bias:** This is the starting point for the distillation rate control. Normally this value does not need to be changed. If you are having problems with distillation rate control contact your local representative for advice.

**Control Mode** shows the current status of the distillation. Control modes include Initializing, Looking for IBP, Looking for volume %, Cooling Down and Shutting Down.

Alarm shows any alarm condition.

At the bottom of the screen is a display of the current system status.

**Pot Temperature** field displays the liquid temperature in the boiling (pot) flask.

**Vapor Temperature** field displays the vapor temperature in the distillation column.

**Vapor AET** field displays the current atmospheric equivalent temperature of the head (vapor). That is, it displays the head (vapor) temperature adjusted for pressure.

**Pressure** field displays the current vacuum level in the distillation apparatus.

**Heat Rate** field displays the current % of the total available wattage being applied to the pot flask.

**Condenser Temperature** field displays the current bath temperature. This is the fluid temperature in the condenser, side arm and chamber.

**Receiver Volume** field displays the current volume of distillate in the receiver.

**Distillation Rate** field displays the current rate of distillate being taken off in milliliters per minute.

- 14. Heat will be applied to the boiling flask according to the Initial Heat Rate set in the distillation program. This heating rate should be as high as possible without causing undue foaming of the sample. The **Run Mode** will be **Looking for IBP.**
- 15. As soon as refluxing liquid appears at the vapor temperature probe, the vapor temperature will begin to rise.
- 16. The IBP (Initial Boiling Point) sensor located just above the receiver will automatically detect and record the first drop as the IBP of the distillation. The **Run Mode** will change to **Looking for 5%**.
- 17. Automatic heat control will take over once the receiver volume has reached 5%. If the Heat % Control mode is chosen the second heating rate will take over at this point. A distillation rate of 6-8 mL/min is desired.
- 18. The computer will automatically record the vapor temperature, time and the pressure at each of the following volume percentage fractions of the charge

- 19. If a sudden increase in pressure is observed, coupled with the formation of white vapors and a drop in the vapor temperature, the material being distilled is showing significant cracking. Discontinue the distillation immediately and record the fact. If necessary, re-run the distillation with a fresh sample at a lower operating pressure.
- 20. Once the end point has been detected the distillation will stop automatically.
- 21. When the distillation has ended the **Run Mode** will switch to **Cooling Down**.
- 22. Lower the flask heater 50 to 10 cm. A fan will automatically blow air on the pot flask to help cool it quickly.
- 23. When the pot cools below 100 °C the vacuum pump will automatically turn off and the system will automatically vent to atmospheric pressure. The **Run Mode** will switch to **Shutting Down.**
- 24. Bring the temperature of the cold trap mounted before the vacuum pump back to ambient temperature. Recover, measure, and record the volume of the light products collected in the trap.
- 25. Remove the receiver and replace with another. Remove the flask and replace with another flask filled with approximately 100 mL of a cleaning solvent such as toluene or cyclohexane. Run a distillation cleaning cycle to clean the unit. At the end of this cleaning run, remove the flask and receiver and blow a gentle stream of air or nitrogen to dry the column.

#### WARNING!

Do not let sample or solvent drip onto the heating mantle. Care must be take to prevent drips onto the heating mantle when the boiling flask is removed. Contaminantion on the heating mantle will damage it and can cause it to fail.

#### WARNING!

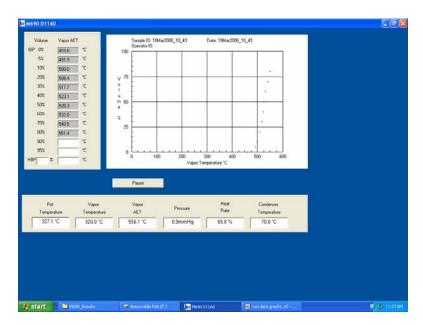
Do not use acetone or other non-petroleum products to clean the system. This will result in damage to critical O-Rings in the system. This may result in serious injury to the user. Use only petroleum solvents such as toluene or cyclohexane to clean the distillation system.

# Other display options during distillation.

By Clicking on the screen, different screens can be viewed during the distillation.

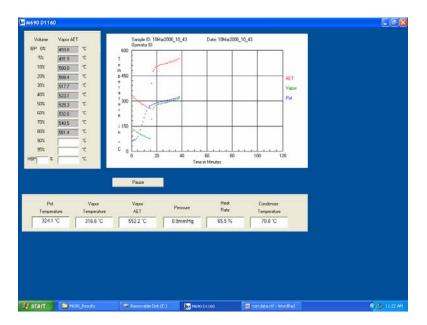
### **Graph Display 1**

Atmospheric Equivalent Vapor Temperature vs. Volume %

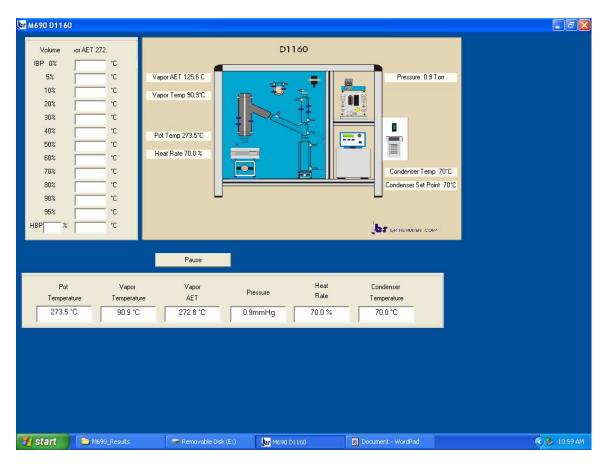


### **Graph Display 2**

Vapor Temperature, Pot Temperature and Atmospheric Equivalent Vapor Temperature vs. Time



### **Process Diagram Display**



## Run Data

Volume	Vapor AE	r	Time	Pot	Vapor	Vapor	Pressure	Condenser		
BP 0%	459.8	T ℃		Temp   °C	Temp   °C	AET °C	Torr	Temp °C		
5%	491.9	τc	000:01:15   000:02:15	63.8 67.4	130.7 125.6	318.1	1.2 0.8	69.0	~	
10%	500.0	°C	000:03:15 000:04:15	69.8 75.8	120.8	312.1	0.9	70.0		
20%	508.4	°C	000:05:15	87.5 104.7	110.3 106.0	298.4	0.9	70.0		
30%		- *C	000:07:15	129.5 154.8	101.4	289.3	0.8	70.0		
40%		- °C	000:09:15	176.1	97.1 93.5	281.0 278.9	0.9 0.8	70.0		
50%		- °C	000.11.15	199.0 220.7	89.7 86.2	273.8	0.8 0.8	70.0		
60%		- °C	000:12:15 000:13:15	239.2 258.4	83.2 80.1	262.6 258.4	0.9 0.9	70.0		
70%		- °C	000:14:15 000:15:15	266.8 271.9	77.1 76.8	257.0 256.6	0.8 0.8	70.0		
80%		°C	000:17:15	274.8 278.4	190.3 251.8	403.6 475.9	0.8 0.9	70.0		
90%		°C	000:19:15	282.4 285.7	261.3 270.7	487.2 498.4	0.9 0.9	70.0		
95%		- °C - °C	000:21:15	288.3 289.8	275.0 277.4	503.5 506.4	0.9	70.0		
*	I	L	000:22:15	291.6	279.9	509.3	0.9	70.0	1	
				_					×	
			Pause							
Pot		Vapor	Vapor		Pressure		Heat	Condenser		
Temper		Temperature	AET	_	Tiessule		Rate	Temperature		
292.3	°C	280.4 °C	509.9 °C		0.9mmHg		49.5 %	70.0 °C		
						1000				
1										

#### Daily

- Follow pre-run checklist
- Carefully document any abnormalities in operation
- Grease Vapor Probe Connection
- Check Condenser Bath Coolant Level
- Check Vacuum Pump Oil Level
- Check all glassware for scratches, chips or any defects and replace immediately
- Always use fresh boiling chips with each run

### Quarterly

• Change Pump Oil

Your D1160 includes all the necessary parts to begin operation. It is recommended that a spare parts kit be purchased for your maintenance stock. If any items need replacement, please contact us or your local distributor.

Description	Part #
Glassware	
200 ml Receiver	K87170-1
500 ml Borosilicate Pot Flask	K87170-2
500 ml Quartz Pot Flask	K87170-3
Vapor Temperature Probe	K87170-4
Adapter	
Electrical	
Heating Mantle Bottom	
115V	K87170-5
220V	K87170-6
Insulating Mantle Top	K87170-7
Temperature Probes	K87170-8
Other	
Anti Splash Guard	K87170-9
Consumables	
Pump Oil	K87170-10
Boiling Chips	K87170-11

Problem	Solution
Poor Distillation Rate Control	Drip trough not in place or not turned so it faces
	forward. Distillation Rate kP too high
Distillation vapor temperature appears too high	Vapor temperature probe holder nut not tightened enough. Temperature probe is getting "sucked" down.
Vapor temperature probe position is set properly but then moved down by itself.	Vapor temperature probe holder nut not tightened enough. Temperature probe is getting "sucked" down.
Level follower moves up and appears "blocked" with a clean receiver in place.	Receiver is not pushed in all the way back into the black plastic base.
Level Follower Eye does not move	Check operation in manual controls
	Make sure lights on stepper motor board are ON (indicates power to board is on)
	Turn level follower screw to move eye by hand
Heating mantle does not heat	Check in manual controls
	Check to see that light on heating mantle relay comes on
Level follower goes up when a run is started.	Lower limit switch is unplugged, blocked or defective.
Distillation rate too high early in the distillation.	Lower the Intial Heat Rate and intial take off rate.
Distillation rate too low early in the distillation.	Raise the Intial Heat Rate and intial take off rate.
Distillation rate too high during the distillation.	Lower the Distillation PID Bias.
Distillation rate too low during the distillation.	Raise the Distillation PID Bias.

**AET:** Atmospheric equivalent temperature is the temperature adjusted for pressure.

Atmospheric pressure: Unit of pressure equivalent to 760 torr (mm Hg).

**Boil up:** The point at which reflux begins in the head of the still. The vapor temperature will rise rapidly as this occurs.

Charge: The sample in the pot flask to be distilled.

**Distillate:** The final product collected after distillation.

**Head:** The "head" is the top of the distillation column where the vapor temperature is measured.

**IBP:** Initial Boiling Point per ASTM D1160

**Pot:** The flask where the sample is boiled.

Takeoff: Distillate removed from the still.

**Takeoff rate:** Amount of distillate removed from the still in a given period of time. This value is usually reported in mls/minute or liters/hour

**Throughput:** The amount of liquid that can be distilled in a given time period.

Vacuum: Removal or evacuation of air and gas from a given space.

**Vapor:** The portion of the sample, which has been transformed from the liquid to the gaseous phase.

**Vapor Temperature:** The temperature of the gaseous phase as measured at the top or "head" of the distillation column. Sometimes this is referred to as the "head temperature".