K87180
Semi-Automatic
Vacuum Distillation System

Operation and Instruction Manual

REV A

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Petroleum Testing & Analysis Instrumentation • Custom Design & Manufacturing

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1 Introduction

The Koehler Model K87180 Semi-Automatic Vacuum Distillation System is the latest design for determining, at reduced pressures, the range of boiling points for petroleum products that can be partially or completely vaporized at a maximum liquid temperature of 400°C according to ASTM D1160 and related test specifications.

This manual provides important information regarding safety, technical reference, installation requirements, operating condition specifications, user facility resource requirements, and operating instructions for the VDS3000 Manual Vacuum Distillation System. This manual should also be used in conjunction with applicable published laboratory procedures. Information on these procedures is given in section 1.2.

1.1 Koehler's Commitment to Our Customers

Providing quality testing instrumentation and technical support services for research and testing laboratories has been our specialty for more than 50 years. At Koehler, the primary focus of our business is providing you with the full support of your laboratory testing needs. Our products are backed by our staff of technically knowledgeable, trained specialists who are experienced in both petroleum products testing and instrument service to better understand your requirements and provide you with the best solutions. You can depend on Koehler for a full range of accurate and reliable instrumentation as well as support for your laboratory testing programs. Please do not hesitate to contact us at any time with your inquiries about equipment, tests, or technical support.

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1.2 Recommended Resources and Publications

1. American Society for Testing and Materials (ASTM)
   100 Barr Harbor Drive
   West Conshohocken, Pennsylvania 19428-2959, USA
   Tel: +1 610 832 9500
   Fax: +1 610 832 9555
   http://www.astm.org
   email: service@astm.org

   ASTM Publication:
   - ASTM D1160: Standard Test Method for Distillation of Petroleum Products at Reduced Pressure

2. International Organization for Standardization (ISO)
   1, rue de Varembé
   Case postale 56
   CH-1211 Geneva 20, Switzerland
   Tel: 41 22 749 01 11
   Fax: 41 22 733 34 30
   http://www.iso.org

   ISO Publication:
   - ISO 6616

3. Japanese Standards Association (JSA)
   4-1-24 Akasaka Minato-ku,
   Tokyo 107-8440, Japan
   Tel: +81-3-3583-8005
   Fax: +81-3-3583-2014
   http://www.jsa.or.jp/

   Japanese Industrial Standards (JIS) Publication:
   - JIS K2254

1.3 Instrument Specifications

Models:	K87180
Electrical Requirements:	220-240V 50/60Hz
Distillation Temperature Range:	Ambient to 400°C (752°F)
Condenser Temperature Range:	Ambient +5 to 150°C
Vacuum Range:	1.00 mmHg to 50 mmHg (0.13 to 6.7 kPa)

2 Safety Information and Warnings

Safety Considerations. The use of this equipment may involve *hazardous* materials and operations. This manual does not purport to address all of the safety problems associated with the use of this equipment. It is the responsibility of any user of this equipment to investigate, research, and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Equipment Modifications and Replacement Parts. Any modification or alteration of this equipment from that of factory specifications is not recommended voids the manufacturer warranty, product safety, performance specifications, and/or certifications whether specified or implied, and may result in personal injury and/or property loss. Replacement parts must be O.E.M. exact replacement equipment.

Unit Design. This equipment is specifically designed for use in accordance with the applicable standard test methods listed in section 1.2 of this manual. The use of this equipment in accordance with any other test procedures, or for any other purpose, is not recommended and may be extremely hazardous.

Chemical Reagents Information. Chemicals and reagents used in performing the test may exhibit potential hazards. Any user must be familiarized with the possible dangers before use. We also recommend consulting the Material Data and Safety Sheet (MSDS) on each chemical reagent for additional information. MSDS information can be easily located on the internet at http://siri.uvm.edu or http://www.sigma-aldrich.com.

Read all instructions carefully before unpacking your system.

Installation and training is provided by Koehler Instrument or a technician trained by Koehler Instrument. 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, call Koehler Instrument.

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 crude oil, crude fractions, petroleum products, and all chemicals.

3 Getting Started

The instructions for preparing the equipment assume that the user is aware of the contents of this document, which lists the warranty conditions and important precautions.

3.1 Unpacking

Carefully unpack and place the instrument and accessories in a secure location. Use extra care while unpacking the glassware set. Ensure that all parts listed on the packing list are present. Inspect the unit and all accessories for damage. If any damage is found, keep all packing materials and immediately report the damage to the carrier. We will assist you with your claim, if requested. When submitting a claim for shipping damage, request that the carrier inspect the shipping container and equipment. Do not return goods to Koehler without written authorization.

3.2 Setup

Electrical Requirements
Three 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.

Manual Cold Trap
The manual cold trap require 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 toluene 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.

4 Descriptions

4.1 Instrument Descriptions

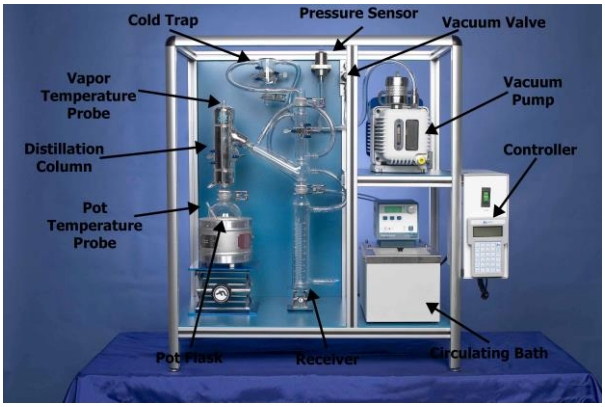


Figure 1: Instrument Descriptions

1. **Vapor Temperature Probe:** This temperature probe measures the actual vapor temperature
2. **Pot Temperature Probe:** This temperature probe measures the temperature of the liquid in the pot flask
3. **Controller:** Controls the distillation system and includes display and keyboard
4. **IBP Sensor:** Automatically detects first drop (Initial Boiling Point)
5. **Receiver:** This is where the distillate is collected
6. **Heating Mantle:** Provides heat to the pot flask
7. **Vacuum Sensor:** Senses the vacuum level
8. **Vacuum Bleed Valve:** This automatically operated valve allows air or nitrogen into the distillation system to regulate the vacuum level
9. **Cold Trap: Filled with dry ice (solid CO2).** The cold trap protects the vacuum pump and pressure sensor from uncondensed gases
10. **Distillation Column:** Column for distilling the sample
11. **Pot Flask:** Flask for distilling the sample.

5 System Preparation

Operational Checks

1. Check the condenser bath level. They should be above the coil inside the bath.

2. Check that the vapor thermometer joint is greased. Lack of sufficient grease on this joint may result in a vacuum leak at low pressures.

3. Check that the pot flask joint is greased. Lack of sufficient grease on this joint may result in a vacuum leak at low pressures. Also grease vacuum adapter and receiver joints.

4. Verify that a clean receiver is in place and empty.

5. Put dry ice in the cold trap.

6. Make sure the vent to atmosphere is closed.

Vacuum System Check

It is very important to verify that the vacuum system is operating properly before each distillation. Check the vacuum pump oil level. It should be near the high mark.

Once the system and been greased, and all joints connected. You can manually start the vacuum sequence without starting an actual program by entering diagnostic 19 on the M690 controller. Turn on the vacuum pump and enter a pressure setting. The system will automatically hold the programmed pressure.

Electrical System Check

1. The M690 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 OFF position.

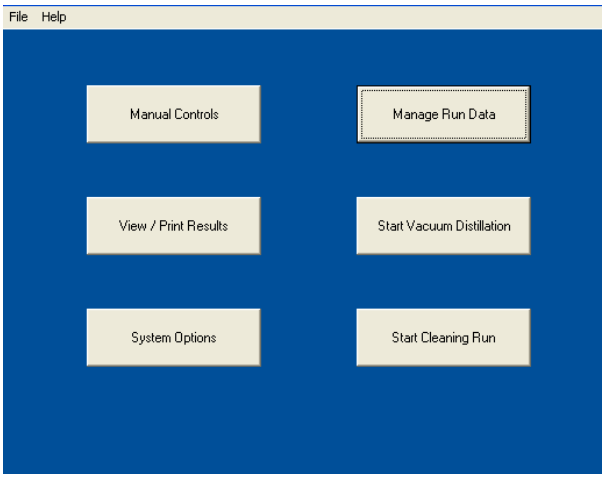
6 Operation

6.1 Software Operation

General

The D1160SA 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



Manual Controls: This area is used for manually operating some aspects of the equipment. Vacuum level, vapor and pot 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.

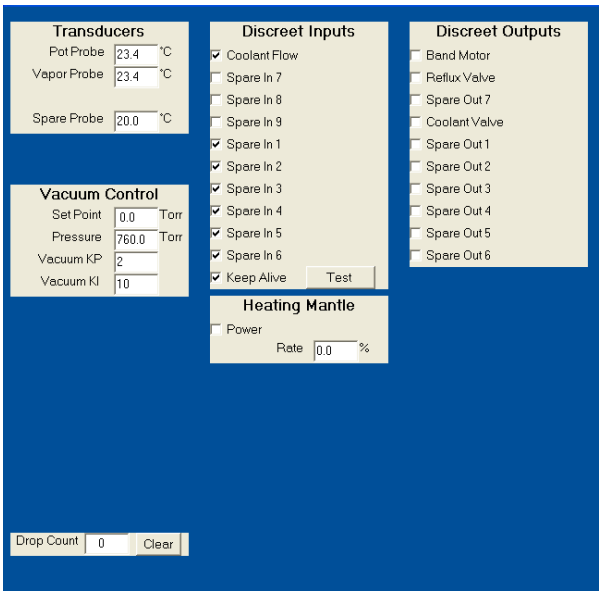
Start Vacuum Distillation: This area is used to start a vacuum distillation run.

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.

Manual Controls

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



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.

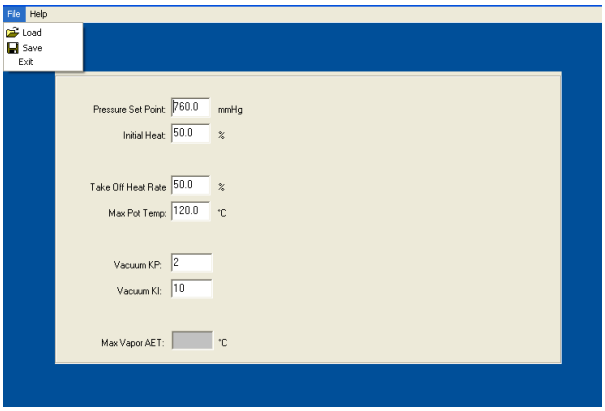
Drop Count displays the number of drops the have been detected by the IBP detector.

The **Heating Mantle** heaters can be turned on by checking the box next to the word

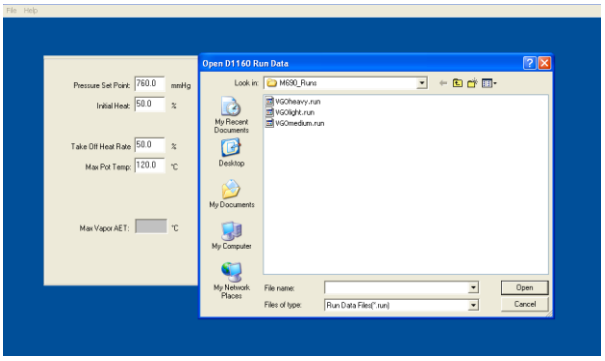
Power. Enter the percentage of the total Wattage desired in the Heat Percent field

Manage Run Data

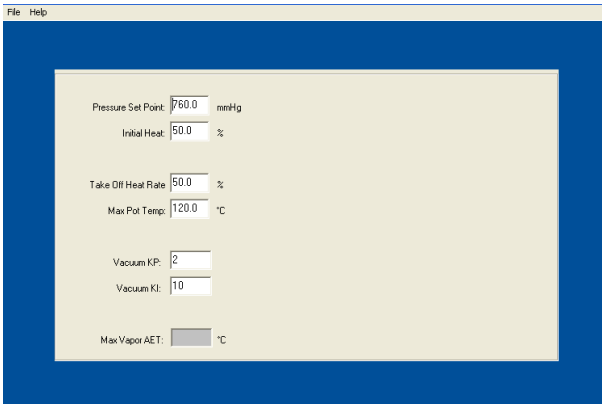
To edit or create a distillation program select LOAD from the File menu.



Select the desired program from the list.



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



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. **Take Off Heat Rate**: This is the amount of heat that will be applied to the pot flask after the first drop is detected. It is used to control the distillation rate. A distillation rate of 6-8 mL/min is desired. Calculate the distillation rate and adjust the heat Take Off Heat Rate so that the distillation rate is 6-8 mL/min.

d. **Maximum Pot Temperature**: This is the maximum pot temperature allowed for this distillation. The default value is 300 ºC. The maximum allowable value is 350 ºC for borosilicate flasks and 400 ºC for quartz flasks. If the pot temperature exceeds 300 ºC for a borosilicate flask during a distillation it should be checked for strain before being returned to service.

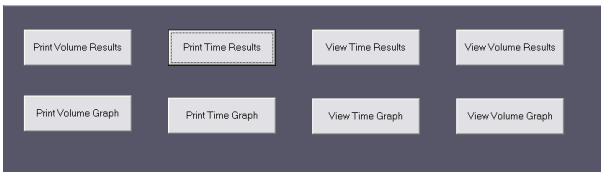
e. **Vacuum KP:** This is the proportional control constant for the vacuum control. The default is 2. If you are having problems with vacuum control contact your local representative for advice.

f. **Vacuum KI:** This is the integral control constant for the vacuum control. The default is 10. If you are having problems with vacuum control contact your local representative for advice.

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.

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.

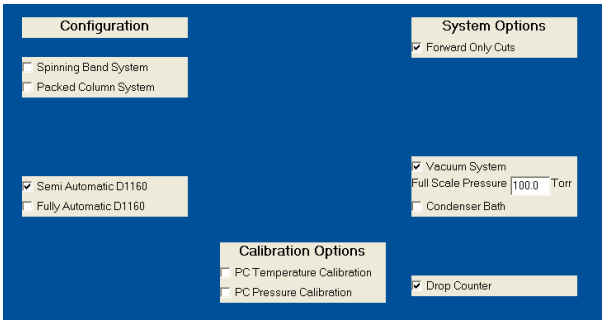
The **View Volume Results** button allows the temperature versus volume data to be viewed.

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.



6.2 Performing a Distillation

1. 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 between 30 to 60 C.

2. 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 if desired.

3. Make sure the anti-splash guard is in the neck of the receiver and if required, the stainless mesh is in the bottom of the distillation column. The mesh is used to break up any foaming of the sample.

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

4. 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.

5. Place a few drops of silicone oil in the bottom of the thermowell of the flask and insert the temperature sensor to the bottom.

6. Turn on the power to the microprocessor controller. The switch is located on the front panel of the microprocessor. 1 is for **on** and 0 is for **off**. When power is applied, the console will sound a brief audible alarm test.

The following screen will be displayed.

Please Wait
Running System Test

Once the system test is successfully completed, the following screen will be displayed.

Select a Command Key

7. If the distillation system is to be used at atmospheric pressure (such as for cleaning), make sure that the vacuum vent valve (below cold trap) is open to atmosphere. If a normal D1160 vacuum distillation is to be performed, close the vent valve and turn on the vacuum pump

8. Diagnostic #19 for the vacuum system can be used to check the equipment for vacuum leaks before beginning a distillation.

9. You may now press **START** followed by one of the stored procedures **(1-20)** and then **ENTER.** (Refer to the section on Store (or Modify) a Distillation Run in Memory to review these prompts.)

10. After entering your response, the console will display the following questions:

A. **Run X**
Run Name
Start Run?
Select YES or NO

B. **Run X**
Boiling Chips in place?
Select YES or NO

C. **Run X**
Is the Pot Fill Plug in Place?
Select YES or NO

D. **Run X**
Is the Pot Secured to the Column?
Select YES or NO

E. **Run X**
Are Head and Pot Probes in Place?
Select YES or NO

F. **Run X**
Are Receivers in Place?
Select YES or NO

G. If the vacuum option is present the following prompt will be displayed.
Run X
Is vacuum pump On?
Select YES or NO

After you have responded to this last question, the distillation begins if all functions are working properly. If malfunction messages appear on the display, refer to the appropriate section of this manual for the cause and solution.

11. The distillation # , the cut #, the head and pot temperatures in degrees Centigrade will all appear in the display window in the following format:
Run X Cut Y
Head Temp = ZZZ.Z
Pot Temp = ZZZ.Z
Pressure XX.X mmHg *If option is active*

12. 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.

13. As soon as vapor or refluxing liquid appears at the head temperature probe, the head temperature will begin to rise. When the head temperature reaches the programmed “open cut” parameter the system will automatically adjust to the programmed “1st heat rate” parameter. With practice, the heat rate settings can be programmed so that the distillate rate falls within the specified rate of 6-8 ml/min quickly. To adjust the heat rate up or down during the run, simply press the “UP ARROW” key on the key pad to increase the heat rate or the “DOWN ARROW” to decrease the rate. Each time you press the key it will change the heat rate 1%. The heat rate will be

displayed on the key pad display each time you press the UP or DOWN key.

14. The IBP (Initial Boiling Point) sensor located just above the receiver will automatically detect and record the IBP of the distillation run. Once detected, the IBP with be displayed on the key pad display, alternating with the current vacuum level. Please make note of the IBP sometime before the end of the run. The IBP will be erased when the "PAUSE" key is pressed at the end of the run.

15. Record the vapor temperature and the pressure at each of the following volume percentage fractions of the charge collected in the receiver: IBP, 5%, 10%, 20%, 30%, 40%, 50%, 60%, 70%, 80%. 90%, 95%, and at the end point. If the pot temperature reaches 400 C, or if the vapor reaches a programmed maximum temperature before the end point is observed, record the head temperature reading and the total volume recovered at the time the distillation is discontinued. The distillation can be discontinued by hitting the PAUSE button.

16. 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 that fact on the run sheet. If necessary, rerun the distillation with a
fresh sample at a lower operating pressure.

17. To stop the distillation run at the end point, simply press the "PAUSE" key. The display will ask "Resume Distillation? Yes or No. Press No. The distillation will also stop automatically if the head temperature reaches the programmed "CLOSE CUT" parameter or if the pot temperature reaches the programmed " POT TEMP TO END RUN" parameter.

18. When the distillation has ended the following will be displayed:
Run X COMPLETED
Head Temp = ZZZ.Z
Pot Temp = ZZZ.Z
Any key to continue

19. Using the lab jack lower the flask heater a few inches and cool the flask and heater with a gentle stream of air or nitrogen.

20. When the pot is sufficiently cool, turn off the vacuum pump and open the vent valve to bring the distillation system to atmospheric pressure.

21. 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.

22. Remove the receiver and replace with another. Remove the flask and replace with another flask filled with a cleaning solvent such as toluene or cyclohexane. Run a distillation at atmospheric pressure to clean the unit (make sure valve is open to atmosphere). There is a "CLEAN" run already programmed into program 20. At the end of this cleaning run, remove the flask and receiver and blow a gentle stream of air or nitrogen to dry the unit.

WARNING! Do not let sample or solvent drip onto the heating mantle. Care must be taken to prevent drips onto the heating mantle when the boiling flask is removed. Contamination 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 alcohol or petroleum solvents to clean the distillation system.

Electrical System Check after the distillation is complete

1. The control unit power should be in the OFF position.

2. The Condenser Bath power switch should be in the OFF position.

3. The Vacuum Pump power switch should be in the OFF position.

Typical Distillation Parameters

Light vacuum gas oil
Enter Run Pressure: 10 torr
Enter Initial Heat Rate: 30%
Enter Open Temp: 40 C
Enter Close Temp: 350 C
Cut 1 Heat Rate: 35%
Add Cut 2: No
Enter Pot Temp to End Run: 400 C

Medium vacuum gas oil
Enter Run Pressure: 1 torr
Enter Initial Heat Rate: 35%
Enter Open Temp: 40 C
Enter Close Temp: 400 C
Cut 1 Heat Rate: 40%
Add Cut 2: No
Enter Pot Temp to End Run: 400 C

Heavy vacuum gas oil
Enter Run Pressure: 1 torr
Enter Initial Heat Rate: 50%
Enter Open Temp: 40 C
Enter Close Temp: 400 C
Cut 1 Heat Rate: 55%
Add Cut 2: No
Enter Pot Temp to End Run: 400 C

Modify a Program During a Distillation

1. Press **MODIFY.** You will be prompted through all of the run parameters and given an opportunity to change them. You are given about 20 seconds between key strokes. If no key stroke is detected within 20 seconds, the display reverts back to the run. You have a total of 2 minutes to make all changes before the display reverts back to the run screen.

Hot Keys

The following functions can be accessed from the keyboard during a run without going
into the modify mode.

1. Increase the mantle rate by 1% increment by pressing the **UP ARROW** key. Decrease the mantle rate by 1% increment by pressing the **DOWN ARROW** key. The heating rate will be changed for the distillation run you are in at the time but is not saved once the run is over.

6.3 Performing a Distillation with PC Option

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 back of the unit. This ensures that the distillate will travel down the back 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.

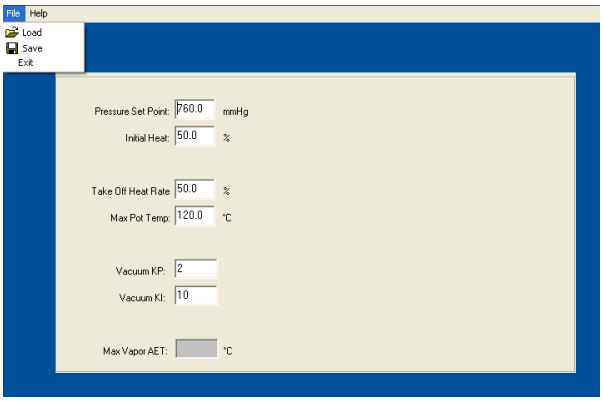
5. Lubricate the spherical joints of the distillation apparatus with suitable grease. Make certain that the surfaces of the joints are clean before applying the grease, and use only the minimum quantity required. Connect the flask to the lower spherical joint of the distilling head, place the heater 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.

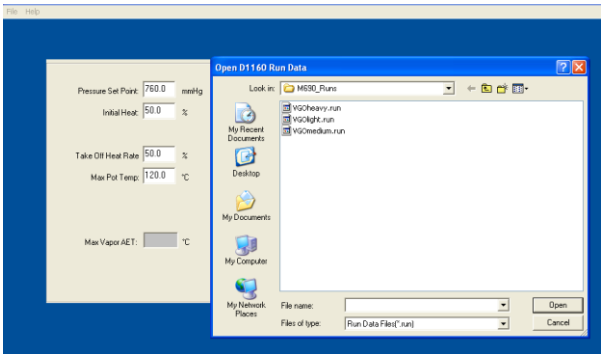
7. Double click the D1160 program icon on the computer. This launches the D1160 distillation program.

8. Select **Start Vacuum Distillation** to begin the distillation.

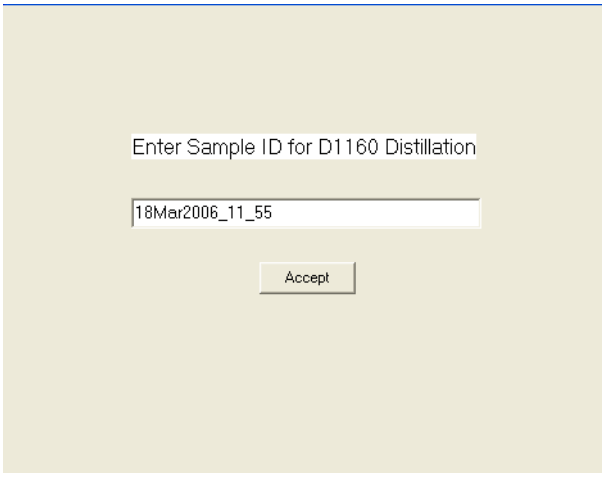
9. Using the file menu choose LOAD



10. Then choose the desired distillation program from the list.

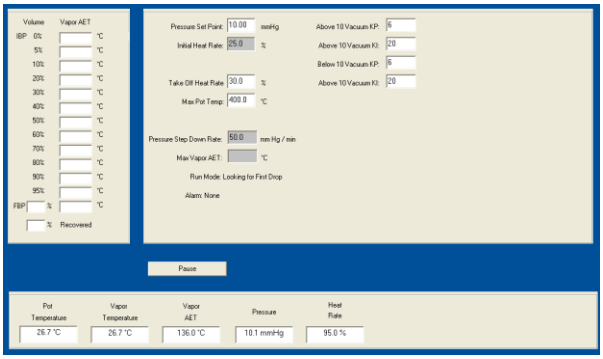


11. Once the program is selected, the distillation program will begin. You will be prompted for a folder name where the results will be stored. The default folder name is the current date and time. This may be changed as desired. The name must have letters and numbers only.



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

13. Once the distillation has started the following screen will be 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.

Take Off Heat Rate: This is the amount of heat that will be applied to the pot flask after the first drop is detected. It is used to control the distillation rate. A distillation rate of 6-8 mL/min is desired. Calculate the distillation rate and adjust the heat Take Off Heat Rate so that the distillation rate is 6-8 mL/min.

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

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 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.

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.

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. The **Take Off Heat Rate** is used to control the distillation rate. A distillation rate of 6-8 mL/min is desired. Calculate the distillation rate and adjust the Take Off Heat Rate so that the distillation rate is 6-8 mL/min.

18. When the receiver volume is equal to 5% select the 5% button. The AET vapor temperature will be automatically stored in the 5% field. Repeat this procedure at each of the following volume percentage fractions of the charge collected in the receiver: 5, 10, 20, 30, 40, 50, 60, 70, 80, 90, 95%. At the final boiling point enter the volume in the volume% field. At the end of the cool down enter the % recovered in the **Recovered** field.

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, rerun 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.

23. When the pot cools below 100 ºC turn off the vacuum pump and vent the system 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 taken to prevent drips onto the heating mantle when the boiling flask is removed. Contamination 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

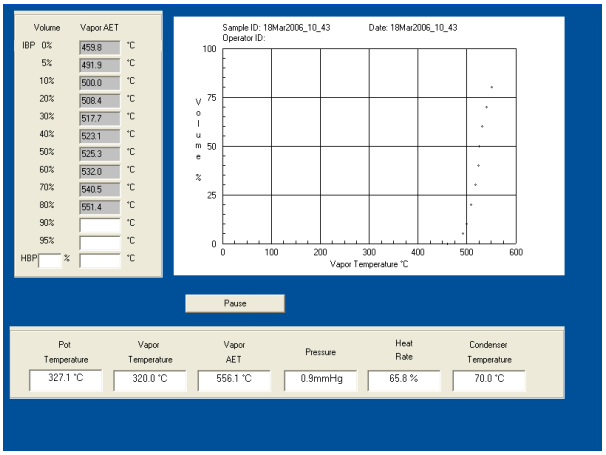
may result in serious injury to the user. Use only petroleum solvents such as toluene or cyclohexane to clean the distillation system.

Other display options during distillation.

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

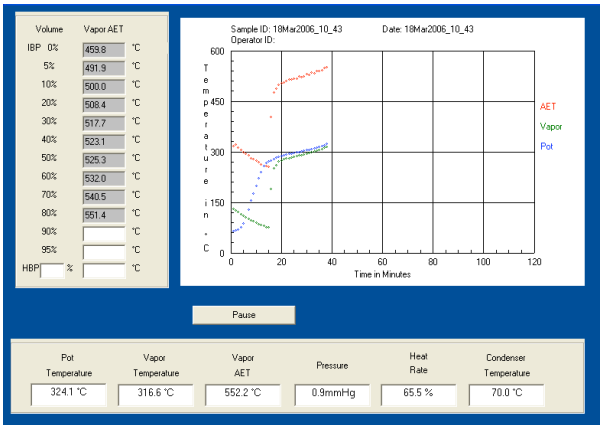
Graph Display 1

Atmospheric Equivalent Vapor Temperature vs. Volume %

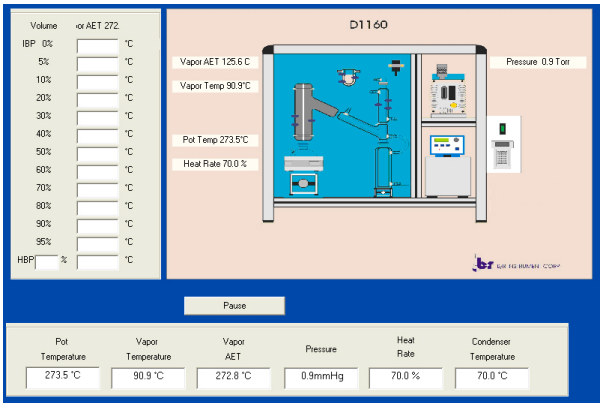


Graph Display 2

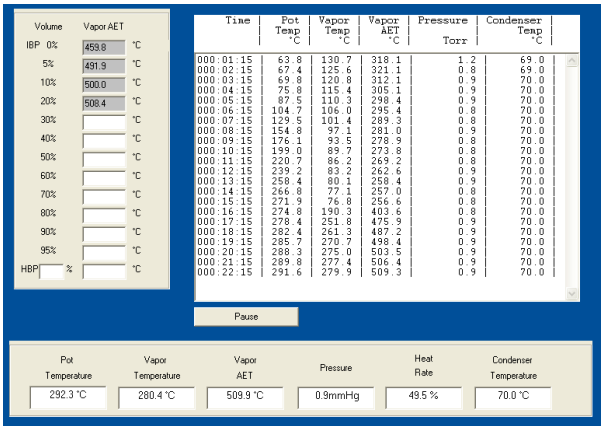
Vapor Temperature, Pot Temperature and Atmospheric Equivalent Vapor Temperature vs. Time



Process Diagram Display Run Data



Run Data



7 Maintenance

WARNING. Disconnect power to the unit before servicing to avoid exposure to high voltages and/or temperatures which may result in personal injury or death. If you have any questions about maintaining your equipment, then please do not hesitate to contact the Koehler technical service department.

7.1 Daily Maintenance

- Follow pre-run checklist
- Carefully document any abnormalities
- Check Condenser Bath Coolant Levels
- 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

7.2 Quarterly Maintenance

- Change Pump Oil

7.3 Zeroing the Baratron

The zero set point for the baratron occasionally needs to be checked for drift. The following procedure is used to make this adjustment. A notebook should be used to record the time and amount of adjustment needed.

1. Attach the baratron directly to the pump inlet.

2. Turn vacuum pump on and allow the vacuum pump to run for fifteen minutes.

3. Turn the zero adjust screw on the baratron until the display for the baratron shows zero.

7.4 Finding a Vacuum Leak

Try each of the following suggestions to resolve the vacuum leak

1. Regrease all joints.

2. Rezero the baratron.

7.5 Replacement Parts

Part Number	Description
K4G-002	200mL Receiver
K4G-007	500mL Pot Flask
KHM500-220VHW	Heating Mantle Bottom
KHM-500T	Insulating Mantle Top
KPRT-1160	Temperature Probes
K4M-002	Anti-Splash Guard
K9C-003	Pump Oil
KBC-20	Boiling Chips

8 Glossary

AET: atmospheric equivalent temperature is the temperature adjusted for pressure.

ACT: actual temperature.

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.

Boil up rate: The amount of the sample that is vaporized and condensed by the condenser. This value is usually given in mls/hour or liters/hour.

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

Distillate: the final product collected after distillation.

Efficiency: a measure of the ability of the still to separate compounds. This is usually referred to in theoretical plates.

Flooding: liquid trapped in the column or the still head by excessive vapors rising through the column. The vapors are rising so fast that the condensate cannot drain into the pot flask. This is usually due to too high of a heating rate on the mantle.

Head: The “head” is the top of the distillation column where the vapor temperature is measured.

IBP: Initial Boiling Point as 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

Theoretical plates: unit used to measure the efficiency of the still. The theoretical plate is the contrivance, which produces the same difference in composition that exists at equilibration of a liquid and a vapor. The more plates there are in a column the greater the efficiency and separating ability.

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 just above the reflux valve. Sometimes this is referred to as the “head temperature”.

9 Service

Under normal operating conditions and with routine maintenance, the K87180 Semi-Automatic Vacuum Distillation System should not require service. Any service problem can be quickly resolved by contacting Koehler's technical service department either by letter, phone, fax, or email. In order to assure the fastest possible service, please provide us with the following information.

Model Number: _____

Serial Number: _____

Date of Shipment: _____

10 Storage

This laboratory test instrument is equipped with electrical components. Storage facilities should be consistent with an indoor laboratory environment. This testing equipment should not be subjected to extremes of temperature and/or moisture.

This equipment was shipped from the factory in a corrugated cardboard container. If long term storage is anticipated, re-packing the instrument in a water-resistant container is recommended to ensure equipment safety and longevity.

11 Warranty

We, at Koehler, would like to thank you for your equipment purchase, which is protected by the following warranty. If within one (1) year from the date of receipt, but no longer than fifteen (15) months from the date of shipment, Koehler equipment fails to perform properly because of defects in materials or workmanship, Koehler Instrument Company, Inc. will repair or, at its sole discretion, replace the equipment without charge F.O.B. its plant, provided the equipment has been properly installed, operated, and maintained. Koehler Instrument Company must be advised in writing of the malfunction and authorize the return of the product to the factory. The sole responsibility of Koehler Instrument Company and the purchaser's exclusive remedy for any claim arising out of the purchase of any product is the repair or replacement of the product. In no event shall the cost of the purchaser's remedy exceed the purchase price, nor shall Koehler Instrument Company be liable for any special, indirect, incidental, consequential, or exemplary damages. KOEHLER INSTRUMENT COMPANY, INC. DISCLAIMS ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY IMPLIED WARRANTIES OF FITNESS FOR A PARTICULAR PURPOSE. Please save the shipping carton in the event the equipment needs to be returned to the factory for warranty repair. If the carton is discarded, it will be the purchaser's responsibility to provide an appropriate shipping carton.

12 Returned Goods Policy

To return products for credit or replacement, please contact Koehler Customer Service with your purchase order number, our packing list/invoice number, the item(s) to be returned and the reason for the return. You will be issued a Returned Authorization (RA) number, which must be prominently displayed on the shipping container when you return the material to our plant. Shipping containers without an RA number prominently displayed with will be returned to the sender. Goods must be returned freight prepaid. Returns will be subject to a restocking charge, the application of which will depend upon the circumstances necessitating the return. Some returns cannot be authorized, including certain products purchased from outside vendors for the convenience of the customer, products manufactured on special order, products shipped from the factory past ninety (90) days, and products which have been used or modified in such a way that they cannot be returned to stock for future sale.

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