# operation and instruction manual



## K24880

## Microscale Continuously Closed Cup Flash Point Tester

## service | performance | technology

**REV K-B** 

1595 Sycamore Avenue • Bohemia, New York 11716-1796 • USA Toll Free: 1-800-878-9070 (US only) • Tel: +1 631 589 3800 • Fax: +1 631 589 3815 http://www.koehlerinstrument.com • e-mail: info@koehlerinstrument.com Petroleum Testing & Analysis Instrumentation • Custom Design & Manufacturing







#### WEEE Directive

#### Background

The goal of the WEEE Directive is to encourage design of environment-friendly products that increase reuse, recycling and other forms of recovery to reduce waste streams and applies to listed Electronic and Electrical Equipment (EEE) and Koehler's equipment falls broadly into Appendix 1A; Section 9 Monitoring and Control Equipment: Measuring, weighing or adjusting appliances for household or as laboratory equipment.

Any associated non-embedded equipment such as Lighting (Saybolt Color) and PCs/Printers also fall under WEEE. If provided with an order these ancillary items must be WEEE compliant. For these and other reasons (printer cartridges are regionalized) the equipment must be supplied through a third party supplier in Europe.

The WEEE Directive applies to electrical and electronic equipment falling under the categories set out in Annex IA provided that the equipment concerned is not part of another type of equipment that does not fall within the scope of this Directive. Annex IB contains a list of products which fall under the categories set out in Annex IA.

http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2003:037:0024:0038:en:PDF

We do not qualify for any of the 10 exemption categories. http://www.dpa-system.dk/en/WEEE/Products/Exemptions

#### Professional use

For equipment defined for 'professional use' local authorities have no role to play. Producers and importers are basically responsible for collection of WEEE recyclables from the professional user and for subsequent management. A separate statement is given cataloging the items that require separation from the equipment along with basic information on subsequent processing or recycling prior to disposal of the equipment.

http://www.dpa-system.dk/en/WEEE/Products/Private-or-professional-use

#### **Responsibility for Registration and Annual Reporting:**

Koehler will not sell directly to end users in the EU and so has no responsibility to register within each EU state and to make annual reports. Koehler declares that this responsibility is born by the importer who is the first level of the distribution chain and is subject to producer responsibility. We will communicate this in writing to our distributor/importers in the EU stating they are responsible to satisfy WEEE registration and reporting requirements in the EU states where they conduct sales activities.

It is illegal to market electrical and electronic equipment covered by producer responsibility without being registered.

http://www.dpa-system.dk/en/WEEE/Producers/Whoissubjecttoproducerresponsibility

#### Product Design

Koehler's designs allow for complete disassembly to a modular level which usually allows for standard recycling. A qualified refrigeration system technician must be consulted when disassembling and de-commissioning any equipment with refrigeration systems.

Koehler's scientific testing equipment is robustly designed to function over a long service life and are typically repaired many times over the course of years rather than being replaced. We believe that re-use and refurbishment is the very best form of re-cycling.

All batteries must be readily removable not soldered in place.



#### **Recycling instructions**

In the event that replacement becomes necessary, we will include instructions, particularized to each instrument that informs the customer of their recycling responsibilities and giving them guidance in doing this. All Koehler equipment has been placed on the market since 13th August 2005 and so Koehler is defined as a "new WEEE producer". As such we must provide information on refurbishment, treatment, and re-use.

Our instrument manual will include this compliance statement and indicate that any collection of materials will be handled by their authorized distributor. In the event that the distributor is unreachable or is no longer a distributor for Koehler Instrument, Co., other arrangements may be made including accepting the materials directly.

Recycling is free of charge. Shipping is the responsibility of the end users. Whether shipping to a distributor or to Koehler directly, safe, properly declared, and labeled packaging and shipping expenses are the sole responsibility of the end user.

#### WEEE Marking



Since Koehler products are subject to the WEEE Directive we must display the WEEE symbol shown above in accordance with European Standard EN 50419 on the equipment. It must be indelible, at least 5mm in height, and clearly legible. If the equipment is too small the mark must be in the product literature, guarantee certificate, or on the packaging. Rules on marking are established in section 49 of the WEEE Order.

Koehler Instrument Company, Inc. c/o RECYCLING 1595 Sycamore, Ave. Bohemia, NY 11716

As a minimum the following substances, preparations and components have to be removed from any separately collected WEEE: - Mercury containing components, such as switches or backlighting lamps (compact fluorescent lamps, CFL),

- Batteries

- Printed circuit boards if the surface of the printed circuit board is greater than 10 square centimeters (about 4 sq in.),

- Toner cartridges, liquid and pasty, as well as color toner,

- Chlorofluorocarbons (CFC), hydrochlorofluorocarbons (HCFC) or hydrofluorocarbons (HFC), hydrocarbons (HC)

- Liquid crystal displays (together with their casing where appropriate) of a surface greater than 100 square centimeters and all those back-lighted with gas discharge lamps,

- External electric cables

- Components containing refractory ceramic fibers as described in Commission Directive 97/69/EC of 5 December 1997 adapting to technical progress Council Directive 67/548/EEC relating to the classification, packaging and labeling of dangerous substances (2),

- Electrolyte capacitors containing substances of concern (height > 25 mm, diameter > 25 mm or proportionately similar volume)

2. The following components of WEEE that is separately collected have to be treated as indicated:

- Equipment containing gases that are ozone depleting or have a global warming potential (GWP) above 15, such as those contained in foams and refrigeration circuits: the gases must be properly extracted and properly treated. Ozone-depleting gases must be treated in accordance with Regulation (EC) No 2037/2000 of the European Parliament and of the Council of 29 June 2000 on substances that deplete the ozone layer (4).



## Table of Contents

1. Introduction	2
1.1. Koehler's Commitment to Our Customers	2
1.2. Recommended Resources and Publications	2
1.3. Instrument Specifications	3
2. Safety Information and Warnings	3
3. Getting Started	3
3.1. Packing List	3
3.2. Unpacking	3
3.3. Setup	4
4. Descriptions	4
4.1. Instrument Description	4
4.2. Input/Output Interface	5
4.3. Miscellaneous Descriptions	5
5. Preparation	6
5.1. Ignition Pin Cleaning	6
5.2. Thermocouple Cleaning	6
5.3. Oven Cleaning	7
5.4. Sample Cup Cleaning	7
5.5. Heating Clean	7
5.6. Connecting External Refrigeration (Optional)	7
6. Operation	8
6.1. Sample Preparation Procedure	8
6.2. Flash Point Measurement	8
6.2.1. Standard Measuring Methods	8
6.3. Custom Measuring Methods	11
6.3.1. Low Temperature Flash Point Measurement (Below 0°C)	11
6.3.2. Scanning Method	12
6.3.3. Determining Flash Point	12
6.4. Parameter Settings	12
6.4.1. Scanning Mode	12
6.4.2. Custom Method	12
6.5. Checking Experimental Results	12
6.5.1. Deleting Experimental Results	13
6.5.2. Copying Data to a USB	13
6.5.3. Printing Experimental Results	13
6.6. System Settings	13
7. Maintenance	13
7.1. Cleaning	14
7.1.1. Oven Cleaning	14
7.1.2. Ignition Electrode Cleaning	14



7.2. Sealability Check	14
7.3. Calibration	14
7.3.1. Adjusting the Pressure Sensor	14
7.3.2. Adjusting the Temperature Sensor	15
7.3.3. Setting Temperature Difference	15
7.3.4. Pensky-Martens Method Offset	15
7.3.5. Rapid Equilibrium Method Offset	15
8. Service	15
9. Storage	16
10. Warranty	16
11. Returned Goods Policy	16



## 1. Introduction

The Koehler Model K24880 Microscale Continuously Closed Cup Flash Point Tester works in the following manner. The temperature of the analyzer is adjusted to at least 18 °C below the expected flash point. The sample is injected into the sample cup. The operator must ensure that the sample and sample cup temperature are both at least 18 °C below the expected flash point (the sample and sample cup may be cooled if necessary). Then the sample cup is raised towards the oven forming a closed test chamber, which is not sealed.

After every ignition, the proper volume of air goes into the test chamber to supply the next ignition with oxygen to burn. The pressure in the unsealed closed test chamber should be ambient atmospheric pressure, except during the short period of air introduction and the time when the flash point is achieved. The pressure increase comes after discharge of air, so each time after the air discharges, the chambers pressure is higher than ambient atmospheric pressure. When the pressure increase exceeds the specified limit, this is recorded as the flash point temperature (which has not been corrected).

This manual provides important information regarding safety, technical reference, installation requirements, operating condition specifications, user facility resource requirements, and operating instructions for the K24880 Microscale Continuously Closed Cup Flash Point Tester. 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 almost 100 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.

Toll Free: 1-800-878-9070 (US only) Tel: +1 631 589 3800 Fax: +1 631 589 3815 Email: <u>info@koehlerinstrument.com</u> http://www.koehlerinstrument.com

## 1.2. Recommended Resources and Publications

- 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 <u>http://www.astm.org</u> email: service@astm.org ASTM Publication:
  - ASTM D6450: Standard Test Method for Flash Point by Continuously Closed Cup (CCCFP) Tester
  - ASTM D7094: Standard Test Method for Flash Point by Modified Continuously Closed Cup (MCCCFP) Tester



## 1.3. Instrument Specifications

Models:	K24880
Electrical Requirements:	100 – 240 VAC, 50/60Hz, 300 W
Temperature Range:	-30 °C to 405 °C (-22 °F to 788 °F) below 0 °C external cryogenic recirculating tank is required
Heating Rate:	2.5 °C/min $\pm$ 0.3 °C or 5.5 °C/min $\pm$ 0.5 °C; can also be customized in the range of 0.5 to 12 °C/min
Temperature Accuracy:	± 0.1 °C
Pressure Range:	0 kPa to 200 kPa
Sample Volume:	1 mL or 2 mL
Ignition Method:	High voltage electric ignition
Stirring Rate:	50 RPM to 300 RPM
Interfaces:	RS232, USB, Ethernet
	8.9 x 12.2 x 16.3 in (22.5 x 31 x 41.5 cm)
Weight:	26.5 lb (12 kg)

## 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 <a href="http://siri.uvm.edu">http://siri.uvm.edu</a> or <a href="http://siri.uvm.edu">siri.uvm.edu</a> or <a href="http://siri

## 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. Packing List

• K24880 Microscale Continuously Closed Cup Flash Point Tester 100-240V, 50/60Hz, 300W

## 3.2. 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.

## 3.3. Setup

**Equipment Placement.** Place the instrument on a firm, level table in an area with adequate ventilation or in a hood. The unit may be leveled by making minor turning adjustments to the feet located at the base of the unit. Please note that Koehler does not supply a level with this equipment. Refer to section 5 for detailed assembly procedure.

**Ventilation.** A fume hood or exhaust system is required when operating the unit. Flammable vapors and/or steam are generated during operation and must not be permitted to accumulate. A canopy-style hood may be used if the height from the top of the unit to the canopy is 5 feet or less. The exhaust blower should have a rating of 1000 C.F.M. or greater.

**Power.** Connect the line cords to properly fused and grounded receptacles with the correct voltage as indicated in section 1.3 or on the back of the unit.



<u>WARNING</u>: For safety, disconnect the power when performing any maintenance and/or cleaning. Do **NOT** turn the power on unless the bath is filled with the proper medium; otherwise, damage may occur to the unit and the warranty will be void.

## 4. Descriptions

## 4.1. Instrument Description



Figure 1. Instrument Descriptions (Front)

- 1. Touch Panel. Display allows the user to run tests and view testing data.
- 2. Touch Pen. To use in conjunction with the touch panel.
- 3. Protective Panel. Protects the inlet and outlet ports when instrument is not in use.



## 4.2. Input/Output Interface

- 1. Ethernet Interface.
- 2. USB Interface. For connection with a mouse or storage device.
- 3. RS232 Interface. For connection with Thermal POS Printer
- 4. Power Interface. For connection to a power supply
- 5. Power Switch.

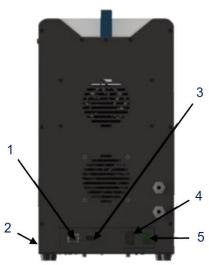


Figure 2. Instrument Descriptions (Rear)

## 4.3. Miscellaneous Descriptions

- 1. 1 mL and 2 mL Sample Cup. Placed within the sample cup holder prior to experiments.
- 2. Sample Cup Holder. Placed in the test chamber along with the sample cup and an appropriate aliquot of test sample.
- 3. Slotted Screwdriver. Used to remove the sample cup from the assembly.
- 4. Cooling Cup. Used to prevent condensation and ice formation on the oven.
- 5. Stirrer. Placed in the sample cup when stirring is necessary.
- 6. Pipette. Used to transport an appropriate amount of test sample to the sample cups.
- 7. Cleaning Brush. Used to clean the ignition device.
- 8. Thermal Printer (Optional). For connection to the RS232 interface to print results when concluding experiments.
- 9. Low-Temperature Cooling Liquid Circulating Pump (Optional). Used to cool the instrument to temperatures below 0 °C.
- 10. External Refrigerating Liquid Tube (Optional). Used to connect the circulating pump to the instrument.



## 5. Preparation

The oven, ignition device and sample cup should be cleaned before every measurement. Please dry them and ensure there is no remainder of any solvent used for instrument cleaning.

## 5.1. Ignition Pin Cleaning

Use the included brush to clean the ignition device surface by eliminating the remaining sample illustrated in Figure 3. Please be careful to keep a distance between the brush and the oven. Then use a tissue to clean the ignition pins carefully (Figure 4).



Figure 3: Cleaning Ignition Device Surface



Figure 4: Cleaning Ignition Pins

## 5.2. Thermocouple Cleaning

Use a tissue to clean the surface of the thermocouple carefully as shown in Figure 5 and be careful not to bend it during the cleaning.



Figure 5: Thermocouple Cleaning



## 5.3. Oven Cleaning

Use a tissue to clean the oven carefully as shown in Figure 6 and ensure the removal of any remaining sample. Please be careful not to scratch the oven. You can use the heat function to do the cleaning if it is necessary.



Figure 6: Oven Cleaning

## 5.4. Sample Cup Cleaning

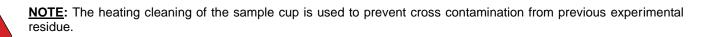
After the sample cup has cooled, use a tissue to clean the sample cup carefully. Take care not to leave any sample residue. Please be careful not to scratch the upper surface.



NOTE: Please ensure the sample cup is not too hot to avoid a burn.

## 5.5. Heating Clean

If the expected flash point of a test sample is 15°C higher than the flash point of the previous sample, heat the empty dry sample cup with the oven until the temperature of the heated sample cup is at least 30°C higher than the expected flash point of the new sample. The specific operation is also explained in Section 6.2.1.



## 5.6. Connecting External Refrigeration (Optional)

The temperature range of the instrument is -30 °C ~ 420 °C. When the indoor environment temperature is 25 °C or lower, the instrument can be cooled to 0 °C without external refrigeration. If you take an external refrigeration measure, the instrument can achieve an even lower temperature. A cryostat, tap water and 5L cryogenic circulator can be used as external refrigerating measures. The flow velocity of the tap water is recommended as 3L/min. Besides water, ethylene glycol can be used as the heat transfer medium of the cryostat. The outlet and inlet on the rear panel are designed for the heat transfer medium shown in Figure 7. By these connectors, the medium is sent to the radiator to cool down to ensure low temperature control.

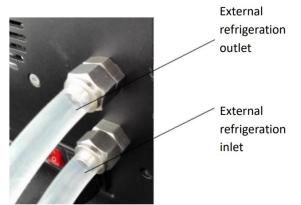


Figure 7: Inlet and Outlet Refrigeration Connections



If the ambient temperature is 20 °C or lower, in order to cool the instrument to -30 °C, the heat transfer medium should be 0 °C. When the ambient temperature is too high, in order to cool the instrument to -30 °C, the heat transfer medium should be -10 °C. If the target temperature of the instrument is 10 °C or lower, the application of external refrigeration measures can improve the refrigerating speed.

## 6. Operation

## 6.1. Sample Preparation Procedure

- 1. According to ASTM D4057 or GB/T 4756, take at least 50mL of sample and place it in a clean container which has been sealed for low temperature preservation.
- 2. A container such as a plastic bottle with high gas permeability is not suitable to store the sample for the high possibility of spreading as volatile materials can spread through the walls of the container.
- 3. Appropriate measures should be taken to avoid the loss of volatile materials which can affect the results. Please do not open the container, unless necessary. Only when the temperature of the sample is 18 °C lower than the expected flash point is it appropriate to pour the sample.
- 4. A sample with high viscosity can be heated to a liquid gradually before the test. The temperature of the heated sample should be lower than the expected flash point.
- 5. Calcium chloride anhydrous can be used to dehydrate a sample which has dissolved water or free water. Use quantitative filter paper or degreasing cotton which is dry and loose to then filter the sample. Heating the sample is allowed, but the temperature of the heated sample should be lower than the expected flash point.



NOTE: If volatile materials are present in the sample, the fourth and fifth steps should be omitted.

## 6.2. Flash Point Measurement

### 6.2.1. Standard Measuring Methods

1. Select the "Maintenance" tab, as shown in Figure 8, select the sampling mode and save. There are two modes: manual sampling and automatic sampling. When set to "Auto off" the sample is put into the test chamber after the temperature of the oven is at the initial temperature. When set to "Auto on" the sample is put into the test chamber at the beginning, and once the temperature is at the initial temperature the test will run automatically.

Measure	Results	Settings	System	Maintenance	About
Mode	0 °C Auto on Auto off	r Function 1 — Ala	rm tion n1 n2 rer	Function 2 Sample hol movemen Motor mover Temp 30 T contro Calibratio User manage	nent 0 C
				2017-07 14:18:5	7-25 TUE i2

Figure 8: Maintenance Interface Screen

- 2. Select the "Measure" interface
- 3. Select the "Method" dropdown menu and choose the test method. Every method has its own interface. If the preset methods do not satisfy you, you can customize your own methods.



- 4. Input the sample name, sample number, sample source, and personnel information. The last 10 values which were typed in will be saved in the drop-down box.
- 5. Set the expected flash point temperature. The instrument calculates the initial temperature and end temperature automatically based on the expected flash point. The initial temperature for the continuously closed cup method and advanced continuously closed cup method is 18 °C lower than the expected flash point, and the end temperature is 26 °C higher than the initial temperature; the initial temperature of Pensky-Martens closed cup method is 23 °C lower than the expected flash point, and the end temperature of rapid equilibrium closed cup method is the same as the end temperature.
- 6. When the initial temperature is below 0 °C, it's necessary to set the cooling mode. The sample refrigerating mode can be set to instrument refrigerating or external refrigerating. If instrument refrigeration mode is selected, the sample should be put into the test room before the instrument refrigerating measurement begins. When set to external refrigeration mode the sample cup with sample should be put into a refrigerator or other external refrigerating equipment for refrigerating at the same time the cooling cup sin placed inside the test chamber and the instrument controls the temperature to the initial temperature. After the instrument reaches the initial temperature replace the cooling cup inside the instrument with the sample cup and sample from the external refrigeration device to run the test.
- 7. After the selection of "Method", corresponding parameters such as Rate (heating rate), Step (ignition frequency), Ignition (ignition interval), P threshold (pressure threshold) and equilibrium time are displayed. The equilibrium time is not usually displayed except for the rapid equilibrium closed cup method. The default pressure threshold is 20 kPa. There is no need to change it in most conditions.
- 8. If the expected flash point is 15 °C higher than the previous sample's flash point, the oven and the sample cup require a heating clean before testing. Set the temperature and time of the clean in the "Settings" interface. The general cleaning temperature is 30 °C higher than the expected flash point. Save the data and go back to the "Measure" interface. Then put the cleaned sample cup into the test chamber and close it. Then press the "Heating clean" button. The temperature of the oven is controlled to the cleaning temperature gradually, then the lifting mechanism raises the sample cup to attach to the oven and keeps the chamber closed during the cleaning period. These heating clean steps are shown on the display screen as shown in Figure 9.



Figure 9: Heating Clean Progress Screen

- 9. After the heating clean, the oven is cooled back to a safe temperature, and the sample cup descends.
- 10. Press the "run" button to begin the test, and the instrument will begin to heat. When the temperature of the oven reaches the initial temperature, the instrument will bring up a popup message (as shown in Figure 10) which says to add the sample cup. If you choose the automatic sampling mode, it will raise the sample cup automatically and run the test.





Figure 10: Insert Sample Cup Notification

- 11. Please insert the proper sample cup volume (e.g. if ASTM D6450, SH/T 0768, SN/T 3077.1 or DL/T 1354 was selected the 1mL sample cup is used. For other methods such as ASTM D7094, the 2mL sample cup is used). Please do not transfer the sample to the hot sample cup, wait for it to be cooled down. Use the pipette to transfer the corresponding sample to the sample cup, and ensure the sample aligns with the graduation line.
- 12. Place the stirrer bar in the sample cup if the sample requires stirring.
- 13. Place the sample cup assembly in the test chamber and ensure it's in the right position, as shown in Figure 11.



Figure 11: Sample Cup Assembly Placement

- 14. Close the test chamber door.
- 15. The instrument raises the sample cup to run the test automatically after closing the test chamber. The present measurement method, sample name, temperature and pressure are displayed on the interface. Press the "Change" button to switch between the parameters interface (Figure 12) and the curve interface (Figure 13).



Figure 12: Parameter Interface



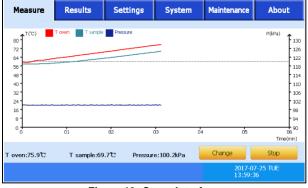


Figure 13: Curve Interface

- 16. The results of the flash point measurement are displayed automatically.
- 17. When the test value is 26 °C higher than the initial temperature, the instrument will display not only the test results, but also a popup message "No flash point was detected within the test temperature range, reset the Expected FP and then try again". If the test value is lower than the expected FP temperature which is set 10 °C higher the initial temperature, the same situation will appear. The recommended expected flash point is this test value.
- 18. If the pressure increase does not exceed the pressure threshold, the instrument will only display the maximum pressure increase and temperature.
- 19. When the measurement is done, the instrument begins to cool down to a safe temperature.

**NOTE:** If you press "Exit", the instrument will stop cooling. Therefore, it is advisable to wait until the cooling is done.

## 6.3. Custom Measuring Methods

### 6.3.1. Low Temperature Flash Point Measurement (Below 0°C)

If the initial temperature is below 0 °C, it is necessary to set the refrigerating mode described in Section 6.2.1 step 1. If there is no refrigerator or samples do not need the pre-refrigeration, then setting the instrument to instrument refrigeration mode will be a good choice. The operation procedure is as follows. Firstly, transfer the sample to the sample cup and put the cup in the test chamber. Then close the test chamber door. Afterwards, press "run". The instrument will bring up a popup message "Instrument refrigerating: The external heat dissipation is opened?" Get the external heat dissipation ready and press "OK". Next the instrument raises the sample cup towards the oven and controls the temperature to the initial temperature to begin the test. This mode avoids the generation of ice on the sample cup and the oven.



**NOTE**: When you are going to test low temperature flash point, please turn on the cryostat or cryogenic circulator in advance for external refrigerating so that the temperature of the medium meets the requirement before the test. After the measurement, please turn off the cryostat or cryogenic circulator in time to avoid condensation or frosting. A large amount of condensate water will be generated in the radiator inside instrument if low temperature circulating liquid is circulated too long. To ensure effective temperature control, high temperature water removal operation is recommended every 2 - 3 tests. Press the "Water removal" button, the instrument will bring up a parameter settings box. Set the control time and cycles next. When water removal is done, press "Exit" to go back to the measurement interface prepare the next test.

When the initial temperature is -15 °C to +10 °C, tap water is recommended as the circulating liquid to prevent the generation of condensate water and long refrigerating time. If there is condensate water from the bottom of the instrument out of the drain hole, please do not worry about it, the test can continue normally. Dumping the instrument is forbidden strictly to avoid damages to the instrument caused by the contact between the water and electric devices. If there is a refrigerator or the sample needs to be precooled, the external refrigeration mode must be selected. At this point, put the sample cup with the sample into an external refrigeration device for refrigerating. The target refrigerating temperature should be lower than the set initial temperature. In this mode, it is necessary to use the supplemented cooling cup to prevent the generation of ice. Next press "run". The instrument brings up a popup message "Instrument refrigerating: The external heat dissipation is opened?". Get the external heat dissipation ready and press "OK". Next the instrument brings up a popup message "Please insert cooling cup and close the door!"



Afterwards, close the test chamber door, the instrument raises the cup automatically and the temperature of the oven is controlled to the initial temperature. When the temperature is ready, the popup message "Please open the door and insert the sample cup" is displayed. At this point, the cooling cup should be replaced with the cooled sample cup with the sample put in.

When the sample cup is ready, the instrument brings up a popup message "Please close the door". The door should be closed as after this notice, and then the sample cup is raised automatically for measurement.

NOTE: If the sample cup is not put into the test room in time, the surface of the oven will ice.

### 6.3.2. Scanning Method

When the flash point of the substance is unknown, the scanning method can be used to estimate the flash point. Higher heating rate and wider control temperature ranges are used in this method.

In the scanning mode, the initial temperature and the end temperature are needed for the measurement. When the initial temperature is below 40°C, the end temperature is recommended to be set to below 80°C. The default parameters (e.g. rate and ignition, etc.) of the scanning method are the same parameters of ASTM D7094. The parameters can be changed as required.

### 6.3.3. Determining Flash Point

Standards ASTM D3828, EN ISO 3679/3680, GB/T 5208, and GB/T 21790 are based on rapid equilibrium closed cup method. This closed cup method tests the existence of the 41 flash point at a specific temperature. The operations are as follows:

- 1. Select the method you want to use, and type in the value of expected flash point (the temperature that you want to test the existence of flash point).
- 2. The default equilibrium time is 1 minute. It can be set in the parameters box if necessary.
- **3.** Press "run". Then the heating begins. When the temperature is controlled to the initial temperature, the instrument brings up a popup message prompting the addition of the test sample. If automatic sampling is used as the sampling mode, the instrument will raise the sample cup automatically at this point.
- 4. After the measurement, the instrument displays results. If flash is tested during the measurement, the results show as normal; if not, the results screen is displayed but with the additional prompt that no flash point was detected.

## 6.4. Parameter Settings

In Standard Mode the parameters vary with the selected test method.

### 6.4.1. Scanning Mode

The preset scanning method of the instrument is based on ASTM D7094 parameter settings. If these parameters do not satisfy you, please build your own scanning method based on the preset scanning method.

### 6.4.2. Custom Method

If the preset methods do not meet your requirements, you can enter the "Settings" interface and set parameters to build your own method. You can customize methods, only if you have the access permission. Though you can change the parameters on the basis of the preset methods, you must rename it (e.g. ASTMD6450-1) because the preset methods are unalterable. Finally, press "save" to save the present custom method.

## 6.5. Checking Experimental Results

- 1. To view results from past experiments, press the "Results" tab.
- 2. The instrument can save at most 5000 experiment results.





**<u>NOTE</u>**: If there are too many results saved on the instrument it may slow down the Results tab. It is advisable to delete experimental results periodically.

## 6.5.1. Deleting Experimental Results

- 1. Select a corresponding experimental record in the experimental results page, and the instrument will fill this area with dark blue
- 2. Press the "delete" button, and the interface brings up a popup message to ensure if you really want to delete this record.
- **3.** Press "yes" button, and the experimental record is deleted.



**NOTE:** To delete multiple results, change "single select" to "multi-select" by pressing the "Single" button. Then select the record to be deleted, press delete, and confirming deletion. It is also possible to sort the "Results" screen to order the results by sample name, operator, test time, etc.

## 6.5.2. Copying Data to a USB

- 1. First insert a USB into the instrument. In the "Results" tab the "Copy to USB disk" button will be displayed.
- 2. Selecting an experimental record and pressing the copy button will then create a copy on the USB.

## 6.5.3. Printing Experimental Results

- 1. Ensure that a printer is connected to the instrument and select the experimental record to be printed from the "Results" tab.
- 2. Press the "Print" button

## 6.6. System Settings

In the system settings interface users can set the Date, Time, Internet IP Address, Units of temperature and Units of pressure.

## 7. Maintenance

- 1. If the maintenance interface cannot be entered, users can operate it before or after the experiment.
- 2. For better experiment control, the instrument functions have multi-level administration. Operators with different jurisdiction can handle the different functions. "Level One" only has the jurisdiction for basic operations, and "Level Two" has all the jurisdiction to operate the instrument and maintain the instrument.
- 3. Operators at different levels have independent passwords. The operator can reset/change the password if they so choose. Press the "User Management" button in the "Maintenance" tab in order to change the password. You also can use the "Factory Reset" function to reset the passwords. After a factory reset, the password for Level One operators is "6666666" and the password of Level Two operators is "888888".
- 4. The function of controlling devices and calibrating parameters can only be used by the user of "Level Two", operation from professional operators is advised.
- 5. To quit the instrument maintenance interface, the selected controlling device will stop running. For example, if you select the alarm, the alarm will start ringing, and then if you switch to the measuring interface, the alarm will stop ringing automatically.
- 6. Press the "Temp control" button to control the temperature of the measuring room to the setting temperature.



<u>WARNING</u>: 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. Cleaning

The instrument requires routine cleaning so as to ensure accurate measurement and results. The necessary cleaning steps are also described in Section 5.

### 7.1.1. Oven Cleaning

The heating cleaning function is used for the oven cleaning, and users just need to press "heating cleaning" in the measurement interface.

When the tested liquids have different properties, there is a need to do a special cleaning of the test chamber. Please make sure that the sample residue has been cleaned, and if necessary, users can use the heating cleaning to prevent cross-contamination. If the contamination happens, the measurement results are suspect. Then the heating cleaning can be done during the measurement.

### 7.1.2. Ignition Electrode Cleaning

Ignition electrode cleaning uses the electric arc cleaning. During the measurement of the standard methods, the electric arc cleaning is done automatically. The default number of times is 3 times.

## 7.2. Sealability Check

- 1. Put the empty sample cup into the test chamber, press "sample cup lift" button in the "Maintenance" interface and then the cup is raised to form a closed chamber. Next select the "introduce air" option to introduce air, and observe the pressure curve of the chamber.
- 2. If the pressure change is in the range of 1.5 2.0 kPa, the sealability is good; if the pressure change exceeds 5kpa, there may be a block in the pressure pipe; if the pressure change is lower than 1.5kPa, the sealability does not meet the requirement so cleaning of the sample cup and the oven is needed to be done or change the sample cup.
- 3. When the pressure pipe is blocked, users need to clean the sample residue as specified in routine maintenance and retest the sealability.

## 7.3. Calibration

When you want to use the instrument to test, you can use the known reference values of flash point to check the instrument. If the difference between the measured flash point value and the reference value exceeds the limit, the instrument should be recalibrated.

### 7.3.1. Adjusting the Pressure Sensor

- 1. Turn off the power to the instrument for 12 hours to make it the same temperature as the environment.
- 2. Prepare a pressure gauge (e.g. mercury manometer) to be placed with the instrument.
- 3. Turn on the instrument, enter the "Maintenance" interface, wait for 10 minutes and then read the pressure value displayed on the instrument and pressure gauge every minute. Read these values 3 times, ensuring the difference is lower than 0.05 kPa every time.
- 4. Calculate the pressure average of the pressure gauge, recorded as P1, calculate the pressure average of the instrument, recorded as P2.
- 5. Calculate  $\Delta P=P1-P2$ .
- 6. Read the constant for pressure calibration from the calibration interface, add it with ΔP, and rewrite it to the instrument.



### 7.3.2. Adjusting the Temperature Sensor

- Open the instrument, apply the thermal conductive silicone grease to the temperature standard instrument (such as secondary standard platinum resistance, diameter: Φ1.0 mm, armor length > 50 mm), and insert it to the measurement hole in the test room.
- 2. Turn off the power to the instrument for 12 hours to make it the same temperature as the environment; "
- 3. Turn on the instrument and enter the maintenance interface; "
- 4. Observe the temperature of the cover and the sample displayed on the instrument and wait until the temperature is stable.
- 5. After the temperature is stable, read the values of the temperature every minute. Read these values 3 times, ensuring the difference is lower than 0.1 °C every time.
- 6. Calculate the temperature average displayed on the temperature standard instrument, recorded as T1, calculate the temperature average of the cover, recorded as T2, and calculate the temperature average of the sample, recorded as T3.
- 7. Calculate  $\Delta$ T1= T1 -T2 and  $\Delta$ T2= T1-T3.
- 8. Read the constants for temperature calibration from the calibration interface, add the constant of the cover with  $\Delta$ T1, add the constant of the sample with  $\Delta$ T2, and rewrite them to the instrument.

### 7.3.3. Setting Temperature Difference

In order to ensure that the heating rate is relatively stable before the ignition, the instrument will start to adjust when the temperature is  $\Delta T$  lower than the initial temperature.

In the calibration page temperature difference field, the test temperature difference is  $\Delta T$ . A higher value brings a more stable heating rate, but the time it takes is longer. Users are not recommended to change this setting.

### 7.3.4. Pensky-Martens Method Offset

This offset is based on the fitting results between the measurement results of the instrument (which is in accordance with Pensky-Martens method) and the Pensky-Martens tests' results using our instrument. Users can use the data using the instrument's Pensky-Martens method and the known Pensky-Martens data to do the fitting by Excel or other tools for obtaining the first-order parameter and the constant term. Type in the first-order parameter and the constant term.

### 7.3.5. Rapid Equilibrium Method Offset

This offset is based on the fitting results between the measurement results of the instrument (which is in accordance with rapid equilibrium method) and the rapid equilibrium tests' results using our instrument. Users can use the data using the instrument's rapid equilibrium method and the known rapid equilibrium data to do the fitting by Excel or other tools for obtaining the first-order parameter and the constant term. Type in the first-order parameter and the constant term finally.

## 8. Service

Under normal operating conditions and with routine maintenance, the K24880 Microscale Closed Cup Flash Point 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:	



## 9. 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.

## 10. 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.

## 11. 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.



## Notes




## Notes
