K441XX
Automatic Non-Woods Metal Noack Evaporative Apparatus

Operation and Instruction Manual

REV A

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Petroleum Testing & Analysis Instrumentation • Custom Design & Manufacturing

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1 Introduction

The Automatic Non-Woods Metal Noack Evaporative Apparatus tests for the evaporation loss tendencies of lubricating oils at temperatures of up to 275°C. The newly designed electrically heated aluminum block allows for testing without the use of hazardous Woods Metal. The Noack tester is equipped with an Electronic regulator allowing for automatic control of temperature and differential pressure. The system is managed by an integrated 6.5" Touch Screen Panel PC by means of the Noack Evaluation Software run by a Windows® based operating System. The Evaluation Software is capable of recording all analytical parameters, allowing for user customizable parameters, methods and result reports as well as printing graphs and test results.

- Conforms to ASTM D5800, Procedure B
- 6.5" Integrated Touch Screen Panel PC
- Integrated Vacuum Pump with automatic electronic control system
- Direct sample temperature measurement via PT100 probe
- Equipped with high resistant Kalrez valve, inlet filter to remove product residuals
- USB port for connection to an external printer and/or external PC
- Storage capacity for more then 60,000 analysis
- CE Marked

This manual provides important information regarding safety, technical references, installation requirements, operating condition specifications, user facility resource requirements, and operating instructions for the Automatic Non-Woods Metal Noack Evaporative Apparatus. 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 D5800: Standard Test Method Evaporation Loss of Lubricating Oils by the Noack Method

2. Energy Institute (IP)
   61 New Cavendish Street
   London, WIM 8AR, United Kingdom
   Tel: 44 (0)20 7467 7100
   Fax: 44 (0)20 7255 1472
   http://www.energyinstpubs.org.uk/

   IP Publication:
   - IP 421

1.3 Instrument Specifications

Model:	K44100 K44190
Electrical Requirements:	115V ±15%, 60Hz 220V ±15%, 50/60Hz
Test Temperature Range:	225°C to 275°C
Measurable Temperature Range:	0°C to 320°C
Temperature Resolution:	0.01°C
Temperature Accuracy:	± 0.2°C
Maximum Ambient Temperature:	35°C
Relative Humidity:	80%
Heating Power:	420W
Repeatability / Reproducibility:	According to ASTM D5800 or better
Dimensions: (L x W x H, in.(cm))	15.75 x 17.72 x 17.72 (40 x 45 x 45)
Net Weight:	48.5 lbs (22kg)

2 Safety Information and Warnings

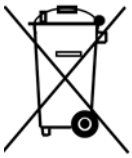
Safety Considerations. The use of this equipment may involve *hazardous* materials and operations. This manual does not purport to address all of the safety problems associated with the use of this equipment. It is the responsibility of any user of this equipment to investigate, research, and establish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

- **DO NOT** allow unauthorized persons to access the analyzer.
- **STAY ALERT!** Do not operate this unit while under the influence of medication, alcohol, or drugs.

Equipment Modifications and Replacement Parts: Any modification or alteration of this equipment from that of factory specifications is not recommended and 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.

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.

Disposal of the Instrument
Please note the crossed out symbol of a trash can on the instrument (please see symbol below). This indicates that the product must be disposed of separate from waste at the end of its operational life. The user that intends to dispose of this equipment must contact the manufacturer to ensure proper disposal.



3 Getting Started

3.1 Packing List

- Automatic Non-Woods Metal Noack Evaporative Apparatus
- Evaporation Crucible for Procedure B
- PT100 Probe for Crucible
- Test Ball (10)
- Nozzle Cleaner
- Crucible Holder
- Protective Gloves
- Hook Wrench
- Pliers
- Operation Software

3.2 Unpacking

Carefully unpack and place the instrument and accessories in a secure location. Inspect the unit 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 Installation

Equipment Placement: Place the instrument on a firm surface in a secure area.

1. Connect the cord cable from the analyzer to a network that conforms to the electric safety specifications

2. Set up glassware as described in the procedure section

3. Turn ON the analyzer

3.4 Additional Accessories Required for Running Tests

Part Number	Description
K44100-1	Glassware Accessory Set ***Includes:*** *2L Glass Bottle (2)* *Rubber Stopper (4)* *Glass Delivery Tubes* *Silicon Connection Tubing*
K44100-2	Stand for glass bottles w/ inclined manometer, 0-50 mmH_2O
K44100-3	Noack Reference Oil, RL223,1L
KLA-DB-KIT	Set of Connectors and Cables
KLA-PT100-CAL	Calibration Decade Box – PT100 Simulator

3.5 Optional Accessories Available

Part Number	Description
K44100-SFW	Noack Evaluation Software
K44100-14	Noack Reference Oil, RL208, 1L

4 Descriptions

4.1 Piping Diagram

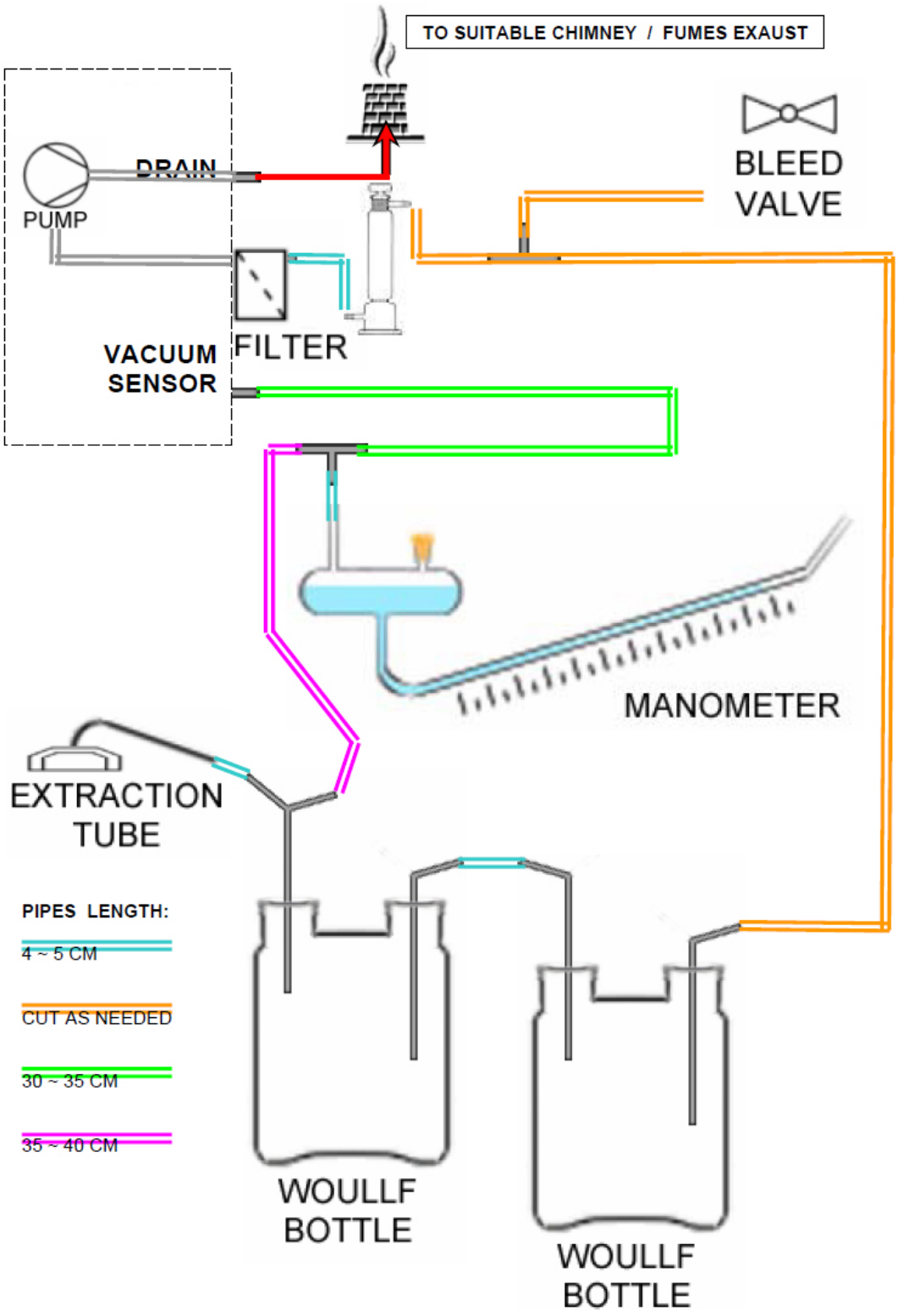


Figure 1: Piping Diagram

4.2 Instrument Controls and Connections

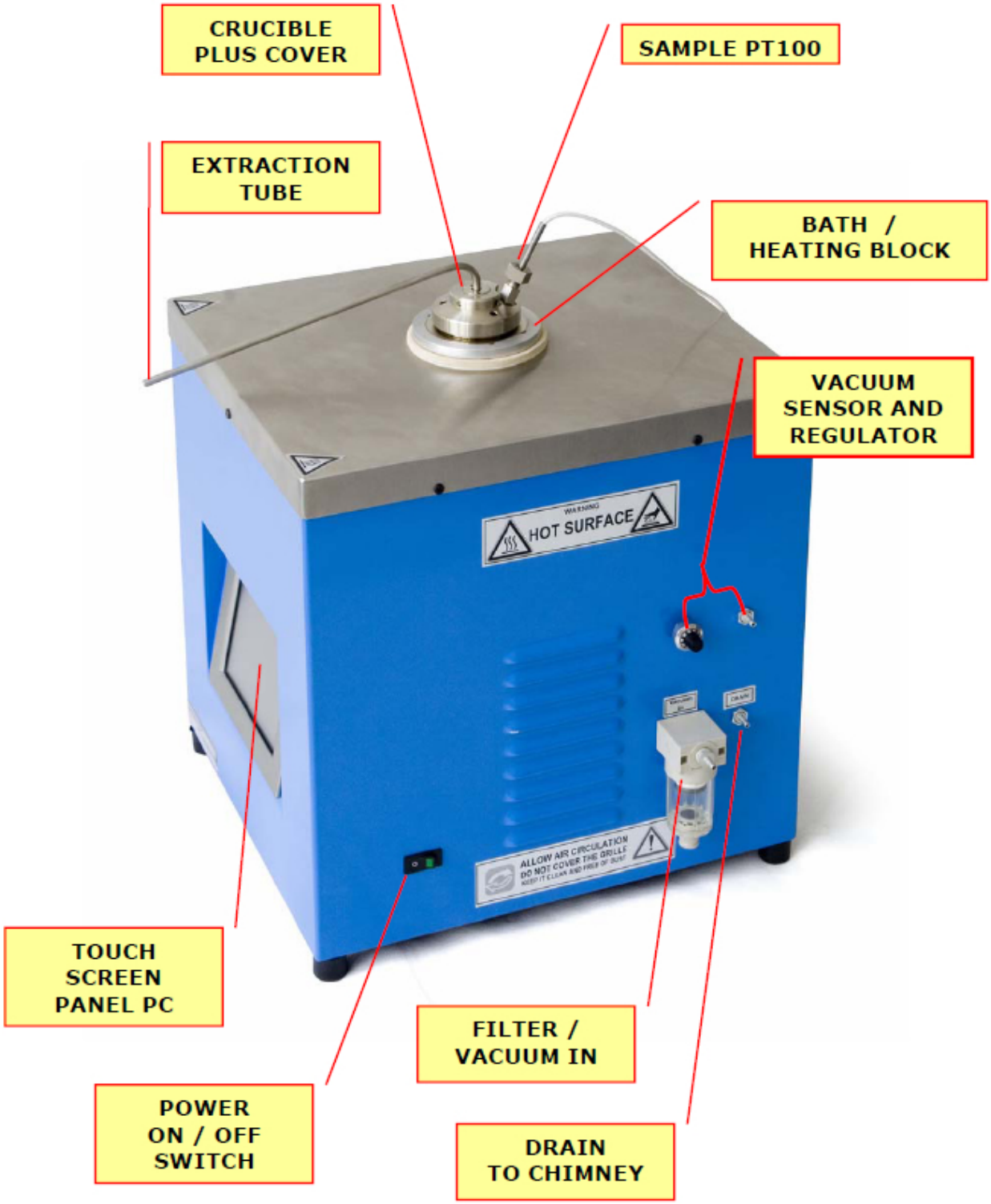


Figure 2: Top / Front / Left Side View

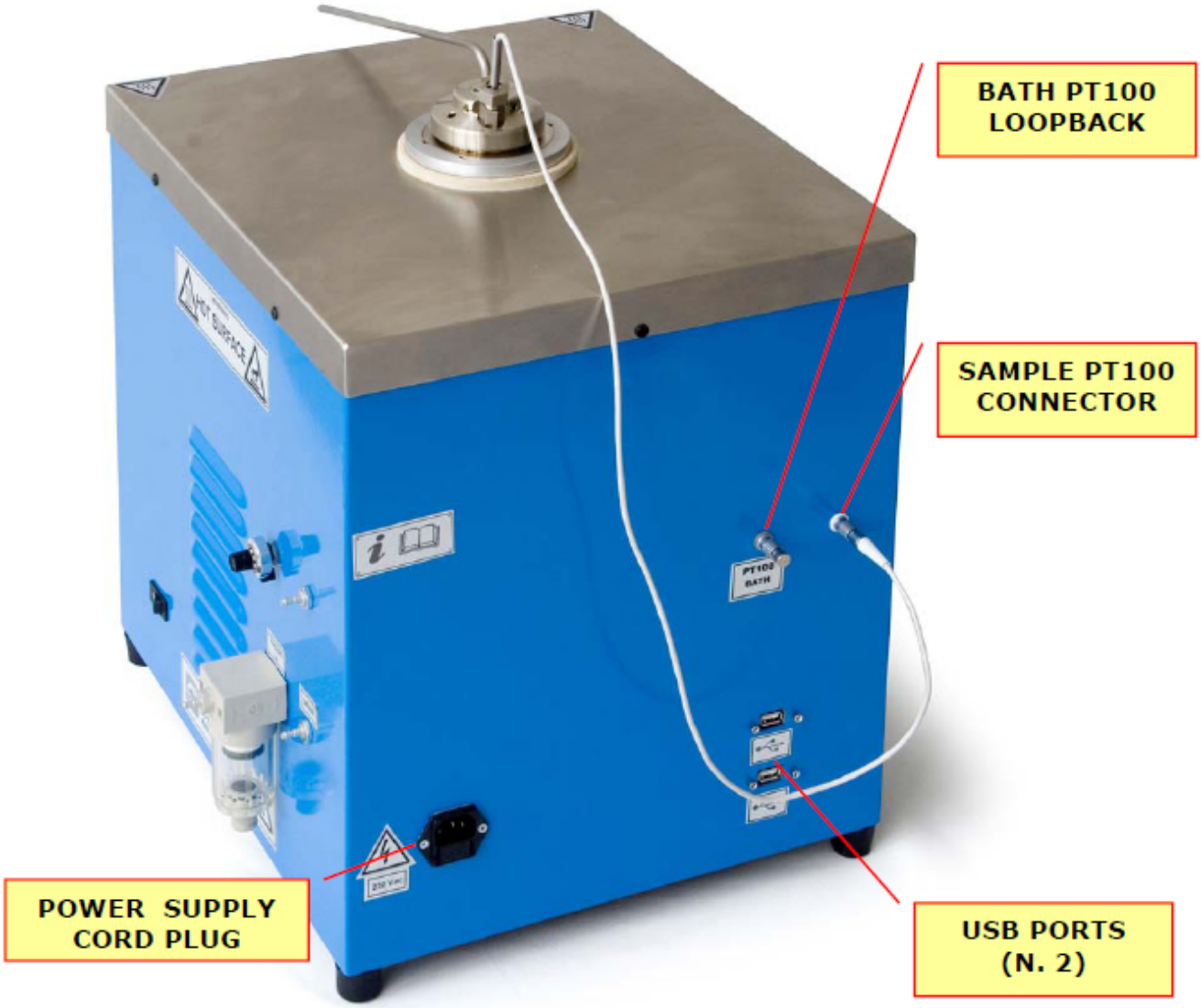


Figure 3: Top / Left / Back Side View

Figure 4: Analyzer Complete with Assembled Glassware Set

5 Instrument Calibration

1. From the main menu screen (See Figure 5 below) select Diagnostic Maintenance.

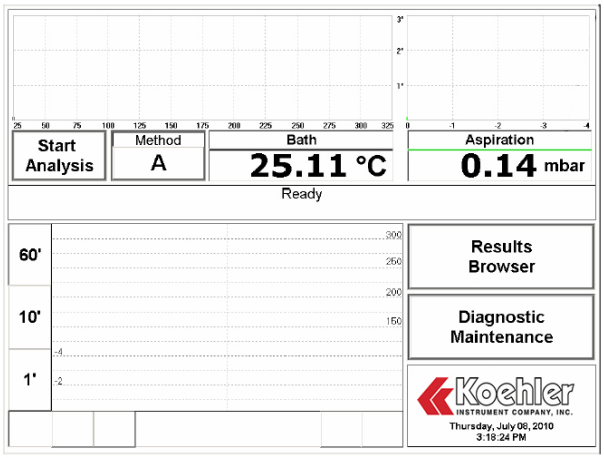


Figure 5: Diagnostic Maintenance Panel

2. In the **Diagnostic Maintenance Panel** (See Figure 6 below) select **Calibration.** This will open the calibration panel.

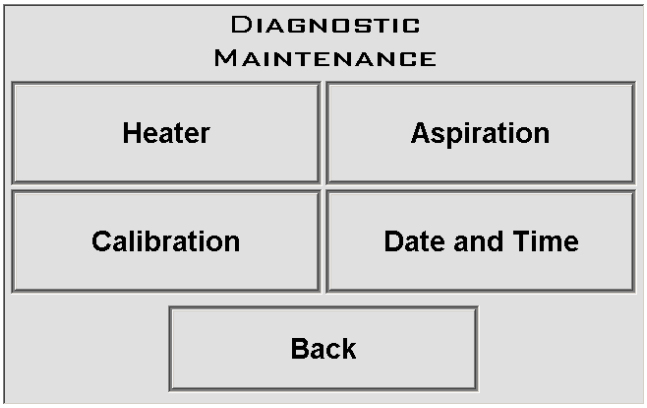


Figure 6: Diagnostic Maintenance Panel

3. At the top of the Calibration Screen (See Figure 7) three buttons will allow the user to choose which probe/ sensor will be displayed. The **Calibrated in** label directly below these buttons shows the selected probe's last calibration date.

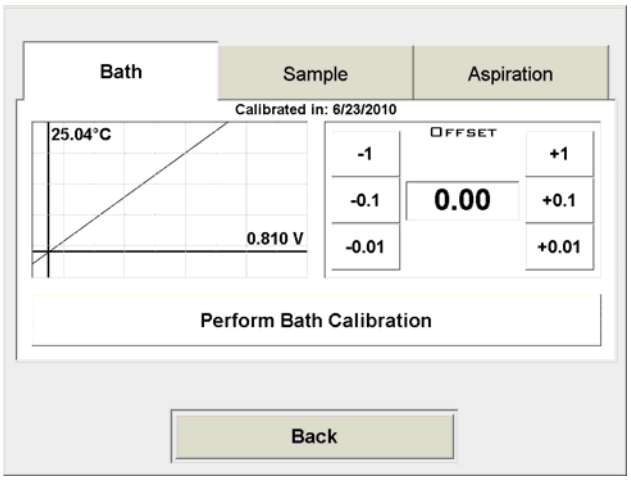


Figure 7: Calibration Screen

4. On the left side of the screen, a graph shows the calibration curve along with the selected probe's value in both °C and volts.

5. On the right side of the screen, the **Offset** field allows for fine tuning of the calibration.

   NOTE: If an offset has been entered, the original calibration curves will be shown as a green line. The black line will move according to the offset value entered in real time.

6. The **Perform Calibration** button starts the guided calibration procedure: to perform a calibration of a temperature probe it is necessary to use a RTD/PT100 Simulator. The recommended RTD/PT100 simulator is the KLA-PT100-CAL Calibrated Decade Box with KLA-DB-KIT Connectors and Cables.

6 Operation

6.1 Summary of Test Method

A quantity of 65 grams of sample is heated in a crucible at 250°C with a constant flow of air drawn through it for 60 minutes. At the end of the test, the sample is cooled and reweighed. The loss in mass of the oil is then determined.

6.2 Analysis Setup Procedure

WARNING: DO NOT INSERT THE CRUCIBLE INTO THE BATH UNTIL THE SOFTWARE PROMPTS THE USER AT THE START OF OPERATION.

1. Press the **Start Analysis** button on the main menu screen (Figure 5) to open the analysis startup panel. See Figure 8 below:

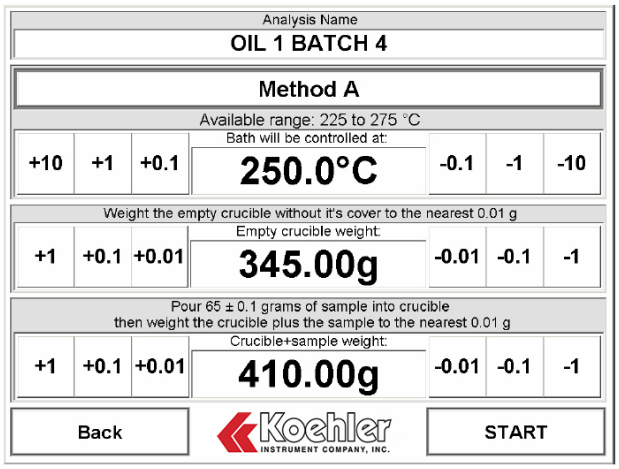


Figure 8: Analysis Startup Screen

2. Touch the **Analysis Name** field to identify the sample. An on-screen keyboard will pop up (See Figure 9 below). The analysis name entered will automatically be listed as the test title in the results browser. This will allow the operator to easily recognize the analysis when using the results browser for review.

Figure 9: On-Screen Keyboard Pop-Up

NOTE: If the analysis name is left blank the analysis will not start.

3. Select the method by touching the **Method** field. Method A is shown in Figure 8 above.

   Method A: Temperature Control via PT100 probe in the Heating Block (Bath)
   Method B: Temperature Control via PT100 probe in the Crucible.

4. Select the set-point temperature for the test based on the selected method. Switching the method will automatically change the temperature to that prescribed by the method.

5. Weigh the empty crucible without the cover to the nearest 0.01g and enter the measured weight.

6. Pour 65g ± 0.1g of sample into the crucible and enter the measured weight to the nearest 0.01g into the dedicated fields.

7. When all of the above parameters are entered, start the analysis by pressing the **START** button.

WARNING: DO NOT INSERT THE CRUCIBLE INTO THE BATH UNTIL THE SOFTWARE PROMPTS THE USER AT THE START OF OPERATION.

8. According to the method and temperature selected, the bath will heat to the prescribed set-point.

   NOTE: The initial heating phase will show the bath's temperature even if method B was selected. During this phase the graph will not display data and the aspiration will remain idle.

9. While the bath is heating, ensure the glassware is correctly set-up as shown on Page Figure 1, Page 6. Verify that the manometer is level and fill with water as needed so that the inclined scale reads zero. **Make sure the bleed valve is fully open.**

10. Once the bath has reached the set temperature an audible alarm will sound and the Crucible Prompt will appear on the screen. See Figure 10:

Figure 10: Crucible / Bath Ready Prompt

11. The **shut off chime** button will appear at the top right corner of the screen when the acoustic alarm is sounded. The volume of the chime is very loud so the operator may want to shut off the chime for the current alarm.

    NOTE: Shutting off the chime will only silence the current crucible alarm. Therefore, future alarms or prompts will still give off an audible chime.

12. As per the on-screen instructions, ensure that the crucible's cover is correctly screwed on the crucible's body.

13. Insert the crucible into the bath and connect the extraction tube to the Y shaped pipe on the Woulff bottle as illustrated on Page 6, Figure 1.

    NOTE: Ensure that the sample PT100 temperature probe is inserted into the crucible and connected to the analyzer even if Method A has been selected.

14. Once the crucible is inserted and the extraction tube is connected, you may touch the screen to start the vacuum and the analysis.

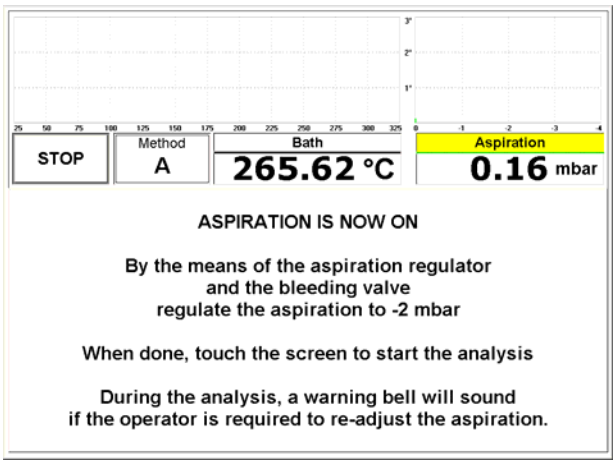


Figure 11: Aspiration Activation Screen

15. The aspiration / vacuum will now be activated. See Figure 11 above. Regulate the aspiration / vacuum to -2 mbar by slowly closing the bleed valve halfway and regulating the vacuum regulator on the analyzer's side in order to reach 20 ± 0.2mm on the inclined manometer (-2mbar on screen).

16. The **Aspiration** label at the right side of the screen will silently flash during this phase until the aspiration reaches its desired setting and is stabilized.

    NOTE: Although the aspiration level should automatically be maintained at -2 mbar it may need to be readjusted at times due to the heating vapor tensions. An acoustic alarm will sound to alert the operator when the aspiration level derives from the prescribed tolerance range. It is imperative the aspiration is readjusted in the shortest time possible.

17. Once the aspiration has been regulated, touch the screen to start the analysis. The analyzer will check if the aspiration is stable, if not, the instructions for its regulation will pop up again to allow the operator to repeat the procedure.

6.3 Test Procedure

1. The **Method** icon is disabled while the analysis is running. If Method B was chosen, the graph will show the Sample PT100 probe temperature. The **Start Analysis** button will become a **STOP** button, allowing the operator to interrupt/abort the analysis.

2. If the analysis is interrupted within 5 minutes from the start, any collected data is discarded and the analysis will not be recorded,

otherwise, even an incomplete analysis running longer than 5 minutes will be recorded.

3. The status bar will describe the progress of the analysis. In the bottom right hand side of the screen the graph will display the temperature data.

4. An alarm will sound along with a full screen notice three minutes before the end of the analysis. See Figure 12 below. Touch the screen to shut off the alarm and close the full screen notice. The status bar will display the countdown to the end of the analysis.

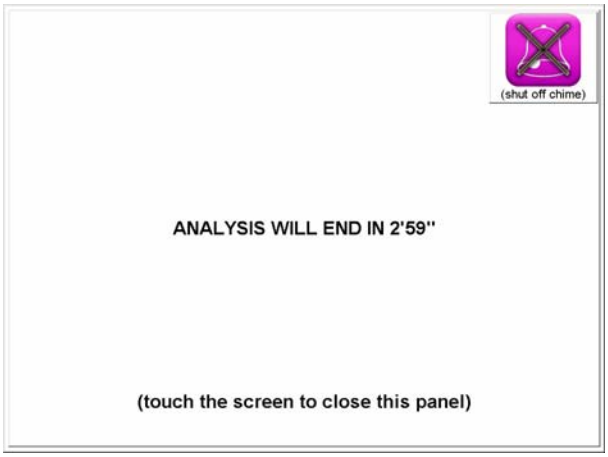


Figure 12: End Analysis Screen

5. As the analysis is approaching the end, prepare 3 cm deep water bath at ambient temperature. This will be used to cool down the crucible; therefore it should be large enough to hold a sufficient amount of water. Sufficient cool down of the crucible will take approximately 30 minutes so accommodate a water bath where the crucible can safely rest without interfering with regular laboratory activities. (A laboratory sink would be sufficient so long as no interference occurs).

6. When the analysis time reaches 60 minutes (the countdown reaches 0), a new alarm will sound. Touch the screen to stop the alarm, then CAREFULLY remove the crucible and place it in the above mentioned water bath. Let the crucible rest in the bath for 30 minutes.

   NOTE: WHILE REMOVING THE CRUCIBLE AND PLACING IT IN THE WATER BATH MAKE SURE THE SAMPLE DOES NOT SPLASH THE CRUCIBLE'S COVER.

7. After 30 minutes, carefully dry the outside of the crucible then, unscrew the cover.
   NOTE: This step should be performed without tilting the crucible. **THE SAMPLE SHOULD NOT SPLASH THE CRUCIBLE'S COVER.**

8. Reweigh the crucible with the remaining (un-evaporated) part of the sample to the nearest 0.01g.

9. In the **Results Browser** enter the final weight. The analyzer will calculate the final result.

6.4 Analysis of the Results

Subtract the empty crucible's weight from the end result in order to maintain the weight of the remaining sample. The result of the following formulas will provide the value of Noack Evaporation Loss (Test Method D5800)

Method A:

$$\text{Evaporation Loss} = \frac{(B\text{-}A) - (C\text{-}A)}{B\text{-}A}$$

Where:

A= Empty Crucible weight
B= Crucible plus Sample weight
C= Crucible plus Sample after 1 hour of heating

Method B:

$[(M_1\text{-}M_2)/M_1] \times 100$

Where:

M_1= sample weight before the test
M_2= sample weight after the test

The final result is given to the nearest 0.1% on both methods

NOTE: The analyzer is able to perform all calculations on the above mentioned formulas, the operator is only required to:

- Note and introduce the empty crucible's weight
- Note and introduce the sample's weight before the test
- Note and introduce the crucible's plus sample's weight after the test

The analyzer automatically gives the result correlation between the two procedures as prescribed in the ASTM D5800 method:

Result on B= 1.030 x Result on A
Result on A= 0.970 x Result on B

6.5 The Results Browser

- Every analysis performed is automatically recorded. The results browser grants access to the analysis archive.
- Use the two arrows on the top of the screen to choose the month/year. See Figure 13 below:
    - The Month's corresponding days are shown in gray if no analysis has been performed.
    - The day is shown in black if the day holds more than one analysis
    - When a day is selected, a green box will appear around that day
- From top to bottom the analysis are enlisted in order of time:
    - For example in the picture below the two analysis where performed at 01:38:14 and 02:18:48 on June 25, 2010.
        - A - sign (as shown in sample 1) means a final weight has not been entered
        - A ± sign (as shown in sample 2) means the sample has been fully completed.
    - The two arrows located on the side allow the operator to select the sample.
    - Press the *Load Analysis Button* (in between the two arrows) to view the analysis.

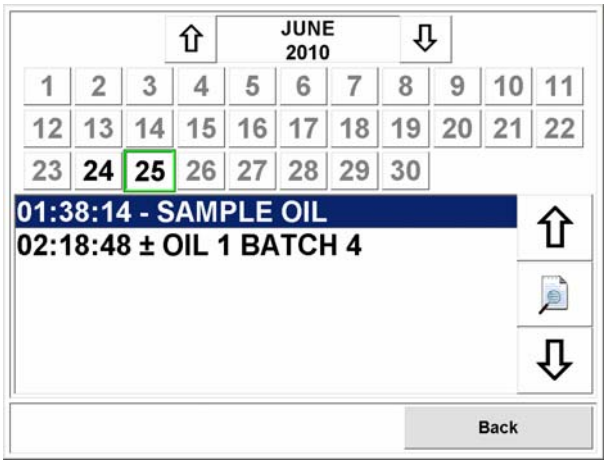


Figure 13: Results Browser Main Screen

If the final weight has not been introduced a question mark will appear in the **Crucible+ Sample Final Weight** Field as shown in Figure 14:

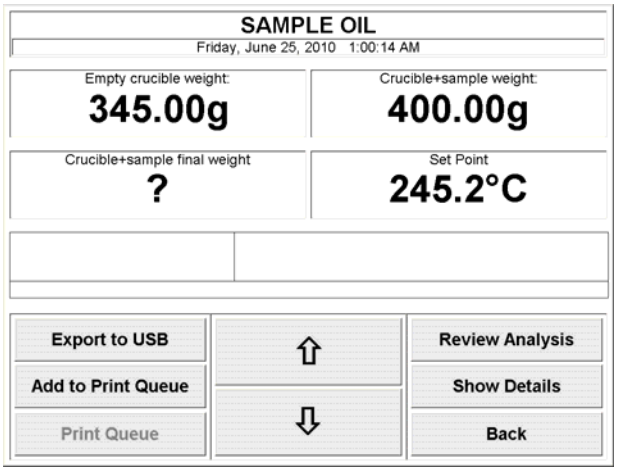


Figure 14: Incomplete Final Weight Screen

To enter the final result, or review any previous parameters, press the **Review Analysis** button (top button on the right hand side). See Figure 15 below:

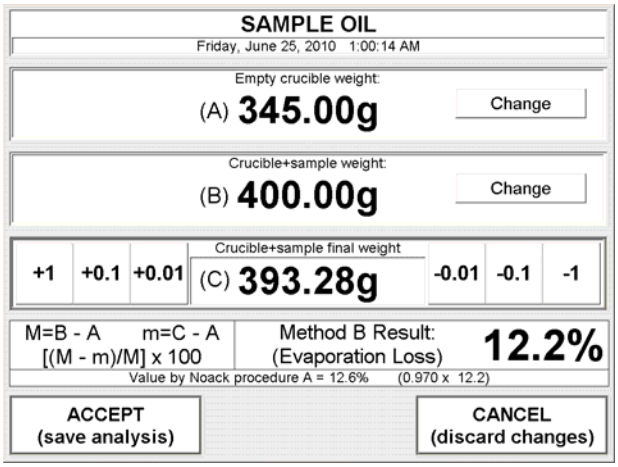


Figure 15: Review Analysis Screen

Once the final weight is entered the result will automatically be calculated according to the method chosen. It is also possible to review/change the other parameters by selecting the **Change** button located on the right hand side of the shown values.

Once the final weight has been introduced press the **ACCEPT** button to confirm, or **CANCEL** to discard the changes and close the panel. Refer to Figure 15 above.

Once the analysis has been reviewed and completed with the final weight, the result browser will automatically show the result and the equivalent result for the other available method. See Figure 16:

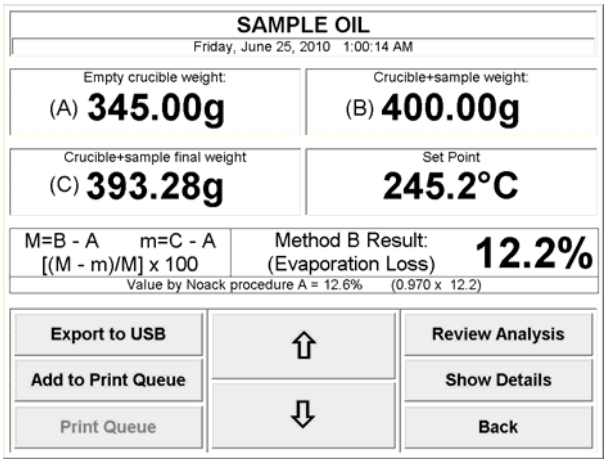


Figure 16: Procedure Result Equivalent Screen

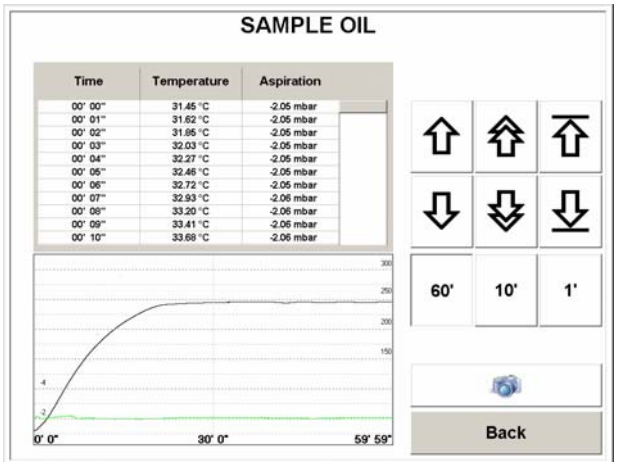


Figure 17: Temperature Graph Result Screen

If a USB Flash Drive is connected, the **Export to USB** button will become available. This allows the analysis to be exported for review on an external USB. If a printer is installed the **Add to Print Queue** and **Print Queue** will be available.

Any USB Windows XP compatible printer can be connected to this analyzer. For detailed instructions on how to install a printer please contact a Koehler Representative as each printer has different characteristics and specific drivers.

Touching the **Show Details** button causes the analysis to reload and the data to be displayed second by second on top of the grid. See Figure 17. Use the buttons on the side to scroll through the data and the 60', 10' and 1' buttons to change the zoom factor on the graph located at the bottom.

If a USB Flash is connected, the **Camera Icon** is shown. Use this button to save a screenshot as a .jpg image.

NOTE: The first panel in the Result Browser will also be updated when a final weight is entered.

The minus sign between the first time and sample name is now a ±. See Figure 18 below.

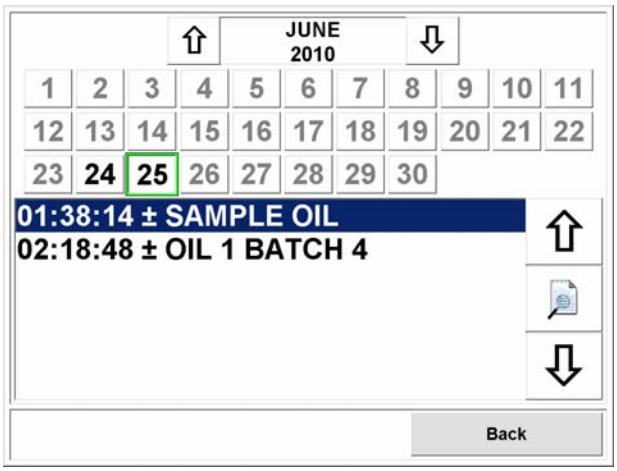


Figure 18: Result Browser Update Screen

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 Routine Maintenance

- Each time a test is run clean the crucible and cover assembly
- Each time a test is run clean the Woullf bottles and pipes
- Each time a test is run clean the heating jacket
- Periodically clean the vacuum filter.

7.2 Replacement Parts

Some instrument parts may need to be replaced. When ordering replacement part(s), please provide the model number, serial number, and product shipment date of your equipment so that we can ensure you will receive the proper replacement part(s).

Part Number	Description
K44100-4	Evaporation Crucible
K44100-5	PT100 Sample Temperature Probe
K44100-6	Silicon Tubing, 2m
K44100-7	Air Filter (for vacuum pump)
K44100-8	Main electronic board for Noack Tester
K44100-9	Hook Wrench
K44100-10	Pliers
K44100-11	Crucible Holder
K44100-12	Test Balls, pk of 10
K44100-13	Nozzle Cleaner

8 Service

Under normal operating conditions and with routine maintenance, the K44100 Noack Evaporation Tester 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 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

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