K47100, K47190
Automated Flocculation Titrimeter (AFT)

Operation and Instruction Manual

Rev B

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1 Introduction

1.1 Background

Historically, asphalts and other heavy oil residua have been modeled as colloidal suspensions in which a polar, associated asphaltene moiety (the dispersed phase) was thought to be suspended in a maltene solvent moiety (the dispersing medium). The extent to which these two moieties would remain in a given state of peptization was thought to be a measure of the compatibility of the suspension according to J. J. Heithaus. This and other similar test methods, along with the classical Heithaus test, are assumed to be a measure of the overall compatibility of a colloidal system as determined by a designated parameter referred to as the state of peptization, P. The value of P commonly varies between 2.5 and 10 for unmodified or neat asphalts. Materials calculated to have low values of P are designated as Incompatible, whereas materials calculated to have high P values are designated as compatible. Values of P may be calculated as a function of two other designated parameters that relate to the peptizability of the asphaltene moiety (the asphaltene peptizability parameter, p_a) and the solvent power of the maltene moiety (the maltene peptizing power parameter, p_o). Values of p_a. and p_o are calculated as functions of the quantities C_{min} and FR_{max}, the values of which are obtained from three experimental variables, the weight of residuum or asphalt (W_a), the volume of solvent (V_S), and the volume of titrant added up to the flocculation point (V_T).

The AFT test method may be used to measure the compatibility or colloidal stability of asphalt and heavy residua by determining the flocculation onset i.e. the point at which asphaltenes just begin to precipitate from a solution of known weight sample prepared in a “solvent” when titrated with a “non-solvent”. Solutions of heavy residua or asphalt, dissolved in toluene, the “solvent”, or other higher solubility solvents (δ = 8.9 H), are prepared in small volume reaction vials (30-mL) and titrated with solvents of lower solubility (δ = 6.9 H, iso-octane, the “non-solvent”). Titrations are performed by delivering titrant, at a constant flow rate via a metering pump, to a stirred test solution. Test solutions, prepared at different initial concentrations W_a/V_S (W_a; weight of asphalt and V_S: initial volume of solvent) in 30 mL “test tube” reaction vials, are temperature controlled by housing the reaction vials in water jacketed beakers temperature regulated with a circulating water bath. The end point of the titration, referred to as the flocculation onset point, is measured based on the change in percent transmission versus time experiments by employing a spectrometer utilized in the capacity of a turbidity detector. This is accomplished by circulating the test solution through Teflon tubing via a second metering pump, to and from a short pathlength flow cell. Time data corresponding to V_T, the minimum volume of titrant required to initiate flocculation onset is determined based on time, t at some maximum detected transmittance value, $\%T_{max}$. Heithaus compatibility parameters p_a, p_o, and P, which relate to colloidal stability, are calculated from initial condition and flocculation onset data (W_a, V_S, and V_T).

1.2 Significance of the Test Method Using the Automated Flocculation Titrator (AFT)

This test method is intended primarily as a laboratory diagnostic tool for estimating the colloidal stability of asphalt, asphalt cross blends, aged asphalt, pyrolyzed asphalt, and heavy oil residuum. Compatibility influences important physical properties of these materials, including rheological properties, e.g., phase angle and viscosity. Compatibility also influences coke formation in refining processes.

1.3 Description of Test Apparatus and Methodology

Samples of asphalt (or a heavy oil residuum), each of different weight, are prepared in specially designed round-bottom reaction vials. Toluene is added to each of the reaction vials at the same volume to dissolve the asphalt (or heavy oil residuum). Thus, each reaction vial contains a solution of different concentration of asphalt (or heavy oil residuum) in toluene. Each of these solutions is then titrated with iso-octane (2,2,4-trimethyl pentane) or some other titrant at a constant titrant delivery rate. A titration is then performed by loading reaction vials, individually, into the AFT apparatus (Figure 1).

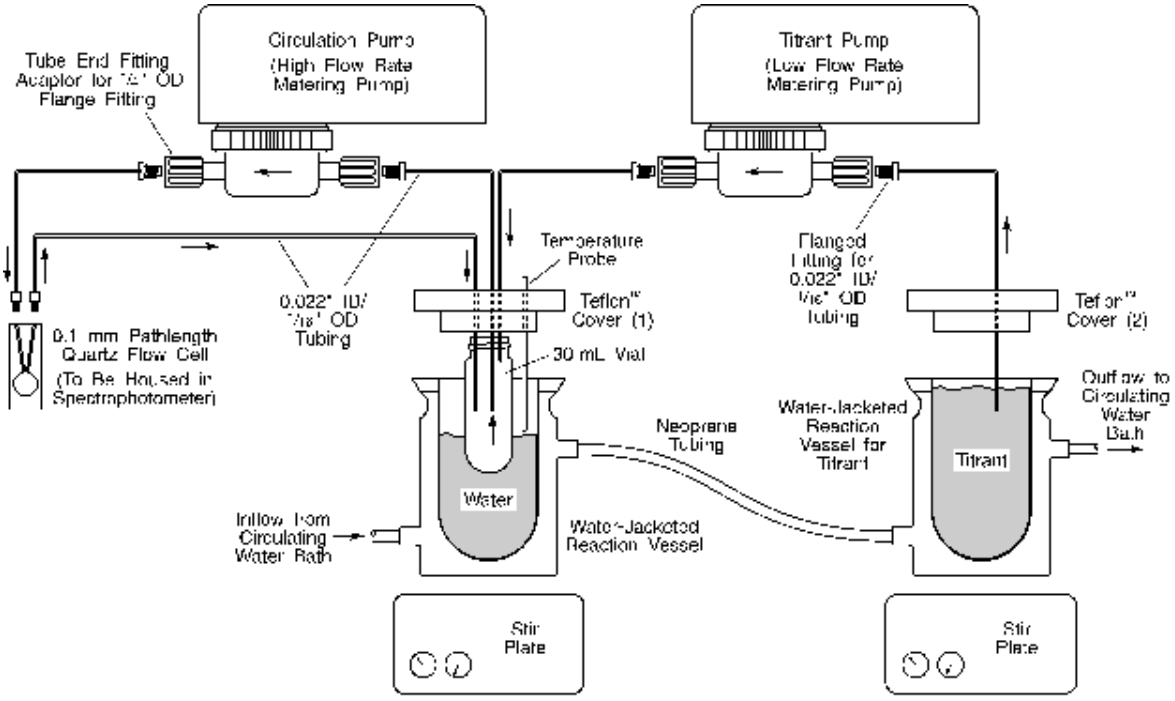
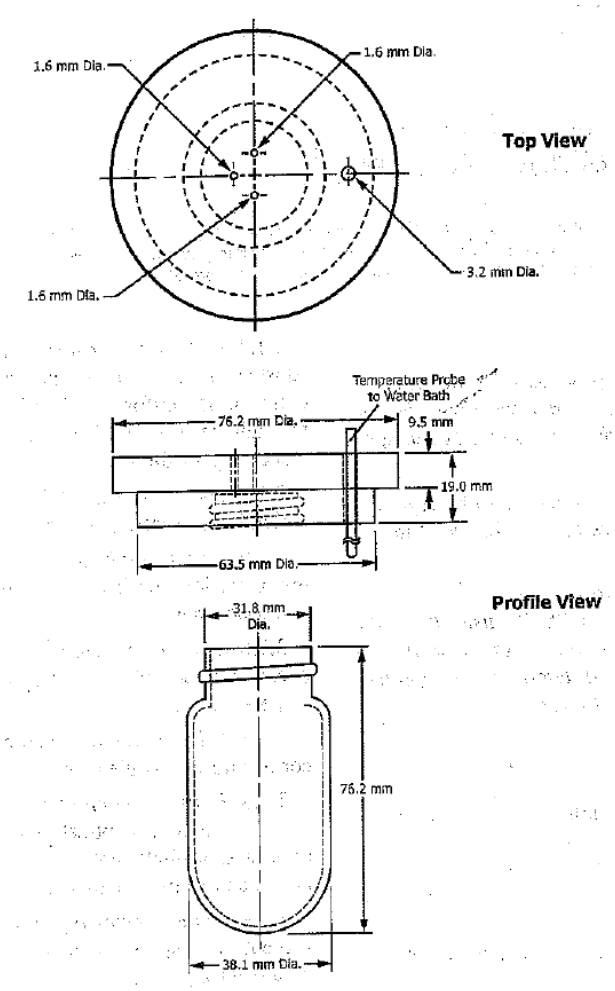


Figure 1. Automated Titration Apparatus

ASTM D6703-01, *2002 Annual Book of ASTM Standards, Road and Paving Materials; Vehicle-Pavement Systems*, Section 4, vol. 04.03. ASTM International, West Conshohocken, PA, 821-829.

Reaction vials are then housed in a water-jacketed reaction vessel (Figure 2) in connection with a sample circulation loop. Teflon tubing is then place into the test solution which leads to a short path length quartz cell housed in a fiber optic UV-visible spectrophotometer. A high flow rate metering pump is then employed to circulate the solution from the reaction vial to the quartz cell and back again to the reaction vial, i.e., the sample circulation loop. Additional Teflon tubing further connects the reaction vial to a low flow rate metering pump, which pumps titrant from another water-jacketed reaction vessel filled with titrant (usually iso-octane). Thus, while the sample solution circulates through the sample circulation loop, titrant is pumped into the sample reaction vial at a constant rate. During this process, an output signal from the spectrophotometer is recorded using a computerized data gathering device. The change in percent transmittance (%T) of detected radiation measured at 740 nm passing through the quartz cell is plotted versus time, t, during which titrant is added to the sample reaction vial at a constant rate.



ASTM D6703-14, *2002 Annual Book of ASTM Standards, Road and Paving Materials; Vehicle-Pavement Systems*, Section 4, vol. 04.03. ASTM International, West Conshohocken, PA, 821-829.

The spectrophotometer output signal detects the onset of turbidity of the sample solution. This is the flocculation onset point, and corresponds to the beginning of the precipitation of asphaltenes from the sample solution. Figure 3 illustrates a typical series of flocculation plots of %T versus t for three test solutions which very by concentration.

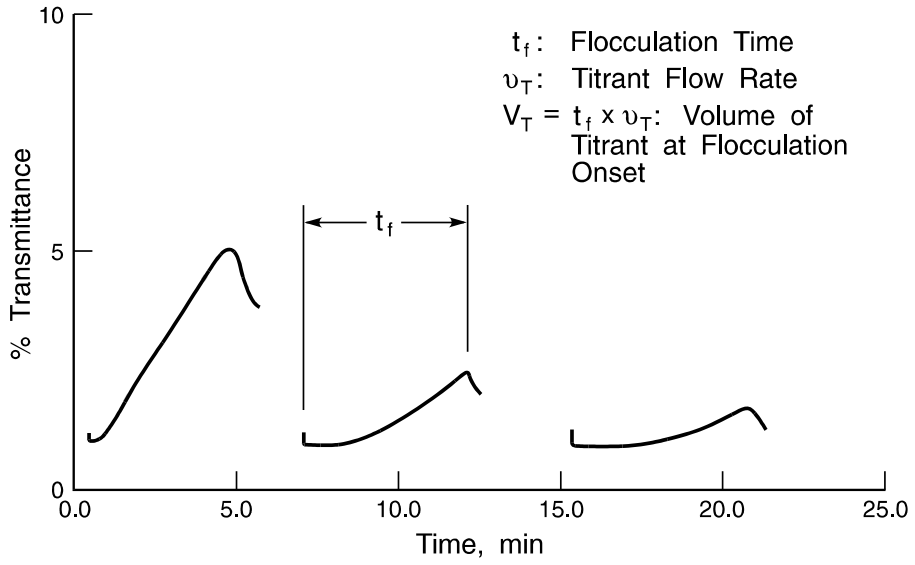


Figure 3. Onset of Flocculation Peaks Measured at Three Successively Increasing Concentrations (Solvent: Toluene, Titrant: Iso-octane)

ASTM D6703-01, *2002 Annual Book of ASTM Standards, Road and Paving Materials; Vehicle-Pavement Systems*, Section 4, vol. 04.03. ASTM International, West Conshohocken, PA, 821-829.

Values of %T increase with time until a maximum value in %T is observed, after which point values of %T begin to decrease. The reason why the curves in Figure 3 exhibit maxima at this point might be due to the following, at the beginning of each titration, %T increases due to dilution with titrant. At the flocculation onset point the formation of flocs of asphaltene particles form to block light and thus cause an immediate decrease in %T due to light scattering effects. The time required to reach the maximum in %T from the onset of titration of a sample is defined as the flocculation time, t_f. When the value of t_f for each sample is multiplied by the titrant flow rate, the titrant volume, V_T, required to cause the onset of flocculation for each sample may then be calculated.

The weight asphalt (or heavy oil residuum) per sample, W_a, the volume of toluene required to dissolve each sample, V_S, and the volume of titrant required to cause the onset of flocculation, V_T, are recorded for each sample solution. Values of three quantities measured for each set of test sample solutions are used to calculate the quantities C (referred to as the dilution concentration) and FR (referred to as the flocculation ratio). C may be derived as $W_a/(V_S + V_T)$. FR may be derived as $V_S/(V_S + V_T)$. Values in C are plotted versus FR for each recorded set of data, i.e., values of W_a, V_S, and V_T (Figure 4). Customarily, C values, measured along the x-axis, and FR values

measured along the y-axis correspond to data points connected by a line that is extrapolated to both axes at x = y = 0. The point at which the line intercepts the x-axis is defined as C_{min}. The point at which the line intercepts the y-axis is defined as FR_{max}. These two values are used to calculate the three Heithaus compatibility parameters, designated p_a, p_o, and P. The parameter p_a, the peptizability of asphaltenes, is defined as the quantity (1 - FR_{max}). The parameter p_o, the peptizing power of maltenes, is defined as the quantity FR_{max} [(1/C_{min}) + 1]. The parameter P, the overall compatibility of the system, is defined as [p_o/(1 - p_a)], or (1/C_{min} + 1).

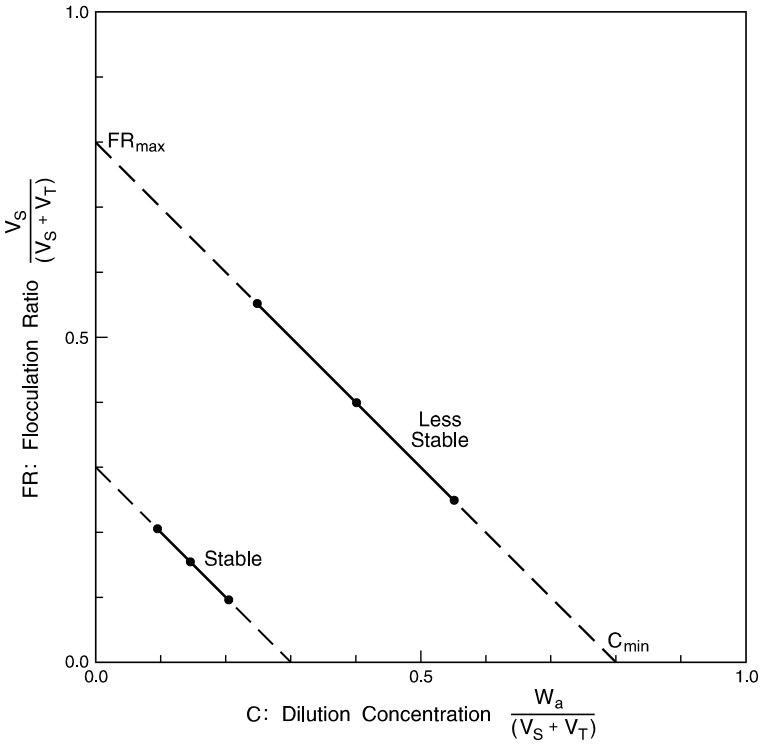


Figure 4. Flocculation Ratio vs. Dilution Concentration for One Stable Asphalt and One Less Stable Asphalt

ASTM D6703-01, *2002 Annual Book of ASTM Standards, Road and Paving Materials; Vehicle-Pavement Systems*, Section 4, vol. 04.03. ASTM International, West Conshohocken, PA, 821-829.

1.4 Unique Features of the AFT

The AFT has been designed as a modular unit. What this means is that the AFT may be expanded to include additional circulation and titration loops to perform multiple titrations at one time. Furthermore, the modular design allows for easy and inexpensive maintenance, repair or replacement of malfunctioning components, if they should occur within the system.

Another unique feature of the AFT is its higher temperature operation feature. This system has been designed to perform titrations in a temperature range between 25 °C and 100 °C.

Recent improvements have also been made to the pump operations and controls. Manual / automatic control switches were added to operate the titration and circulation pumps. This allows the pumps to be operated manually for cleaning and priming without the need to operate the instrument under PC control. It also allows the pumps to be stopped if any issues arise during testing.

A reverse flow push button has been added to the circulation pump section. This is to aid in un-clogging the lines if they become clogged during the test. The AFT Control Panel is pictured in **Figure 5** below:

Figure 5. AFT Control Panel Descriptions

Figure 6. AFT Control Panel Descriptions - Left Side Close Up

Titration Pump Control. Switch has three (3) positions: **ON** - Manual Mode. **OFF** - Pump Off. **AUTO** - Automatic Mode allows the PC to control the pump action.

Stirrer Speed. Controls the speed of the magnetic stirrer. Turning the knob to the down position will turn off the stirrer.

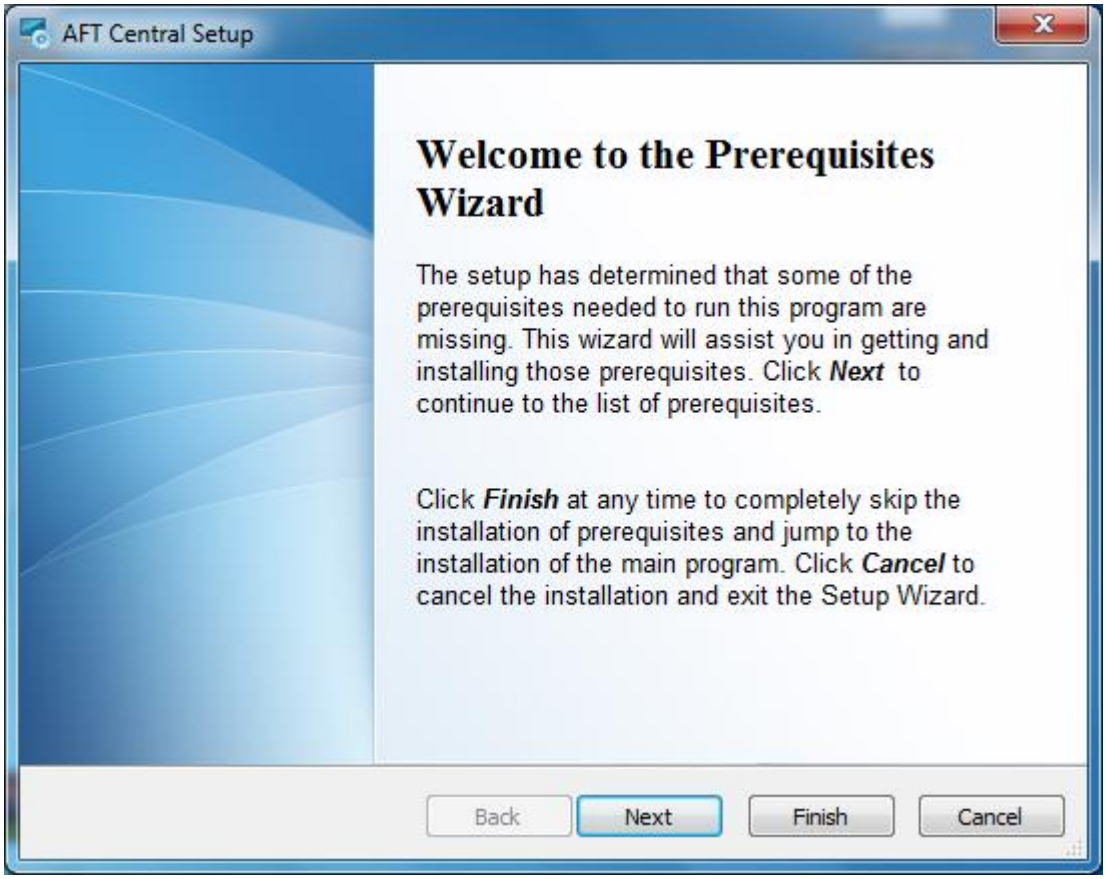
Power Switch. Controls the power to the entire unit.

Figure 7. AFT Control Panel Descriptions - Right Side Close Up

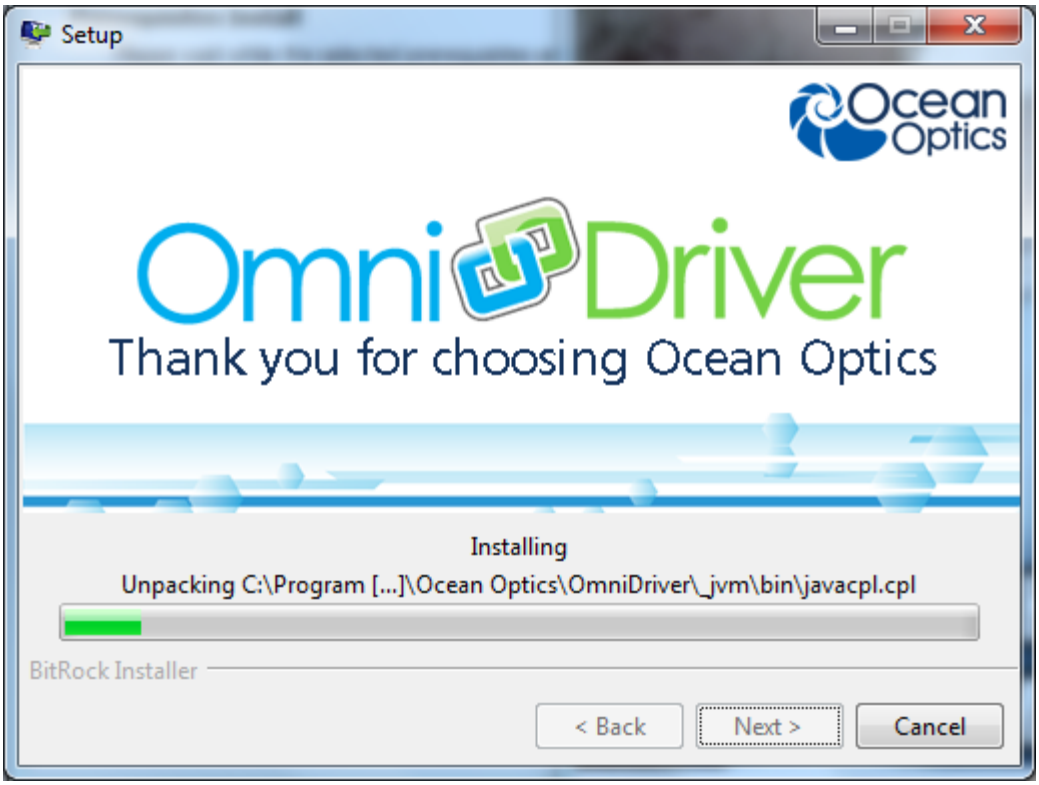
Stirrer Speed. Controls the speed of the magnetic stirrer. Turning the knob to the down position will turn off the stirrer.

Circulation Pump.
- **SPEED**: Regulates the circulation pump speed
- **REVERSE**: Reverses the pump direction when it is running. It is a momentary push button to temporarily reverse the pump action.
- **CONTROL**: Switch has three (3) positions: **ON** - Manual Mode. **OFF** - Pump Off. **AUTO** - Automatic Mode allows the PC to control the pump action.

2 Software Installation (AFT Central v. 1.5.0 for Windows XP)

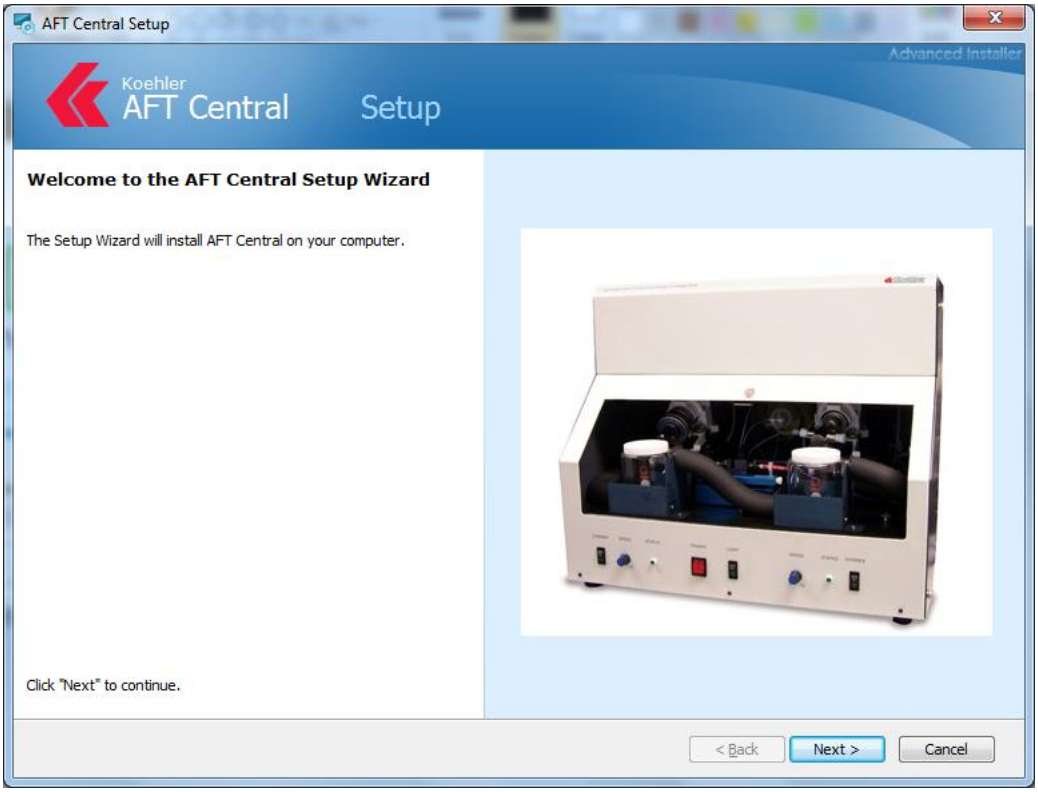


Prerequisites installer wizard which will install Omnidriver and other drivers needed for the software. Click Next to Continue.

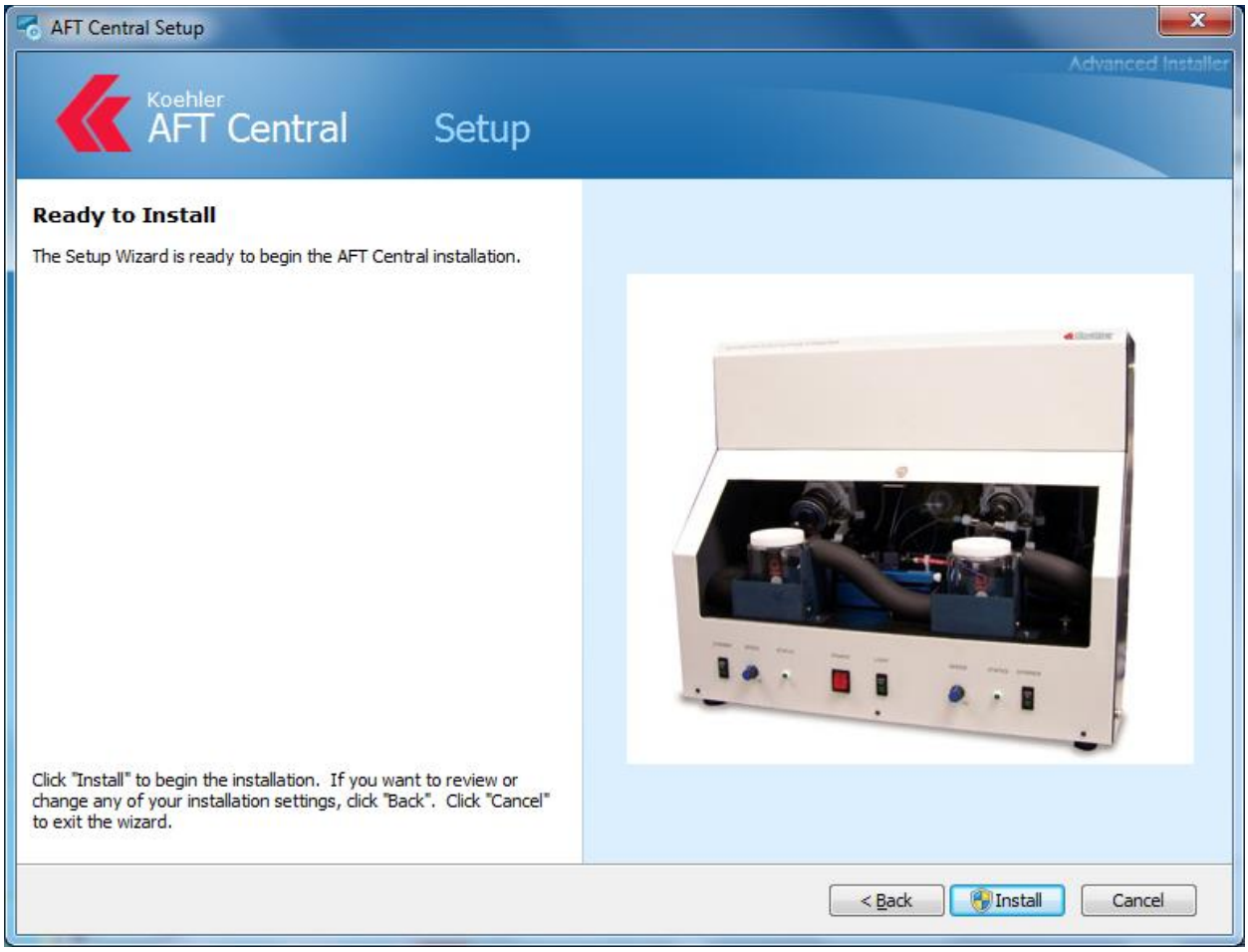


Installing Omnidriver software for spectrometer and light source communication

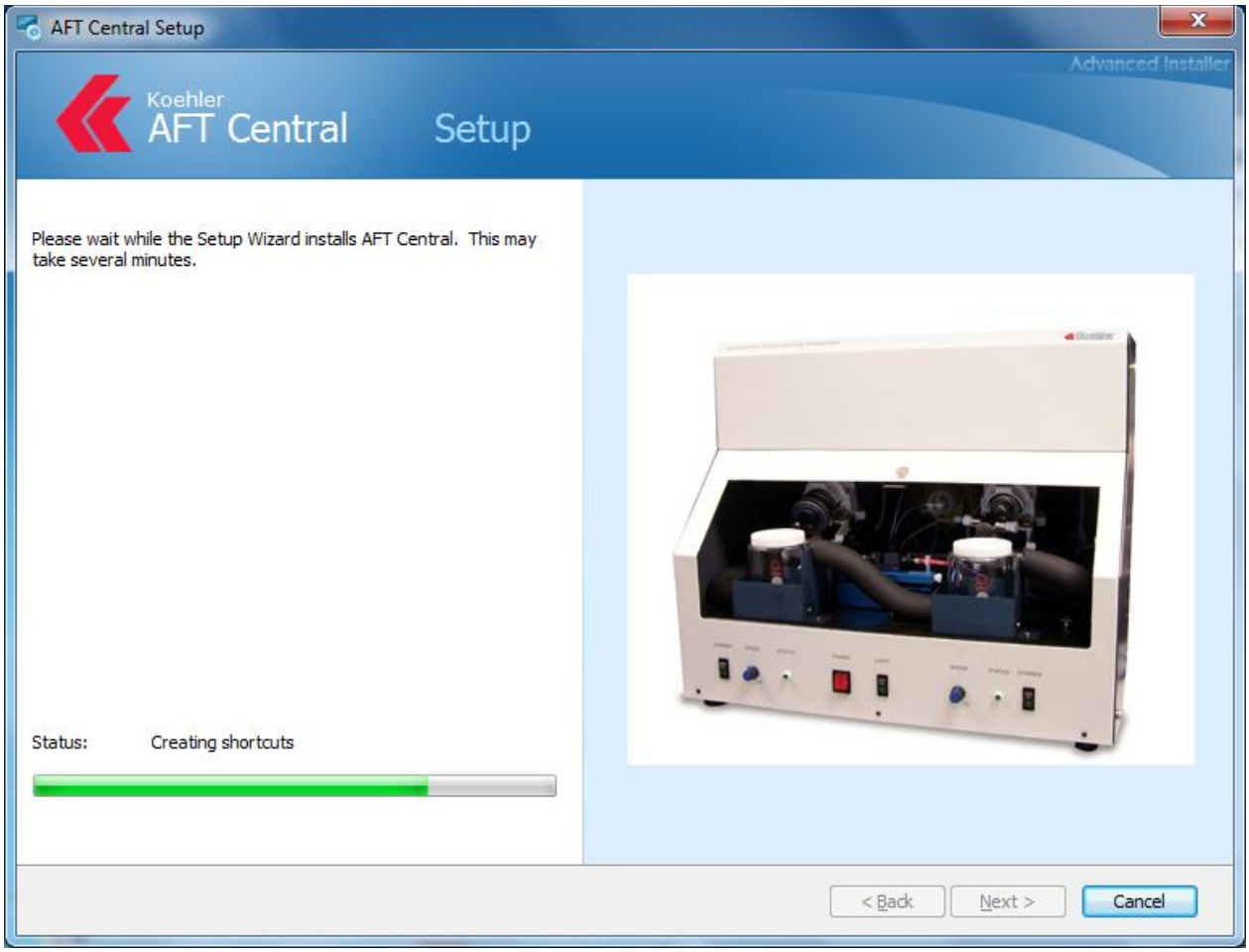
Spectrometer Omnidriver Installer



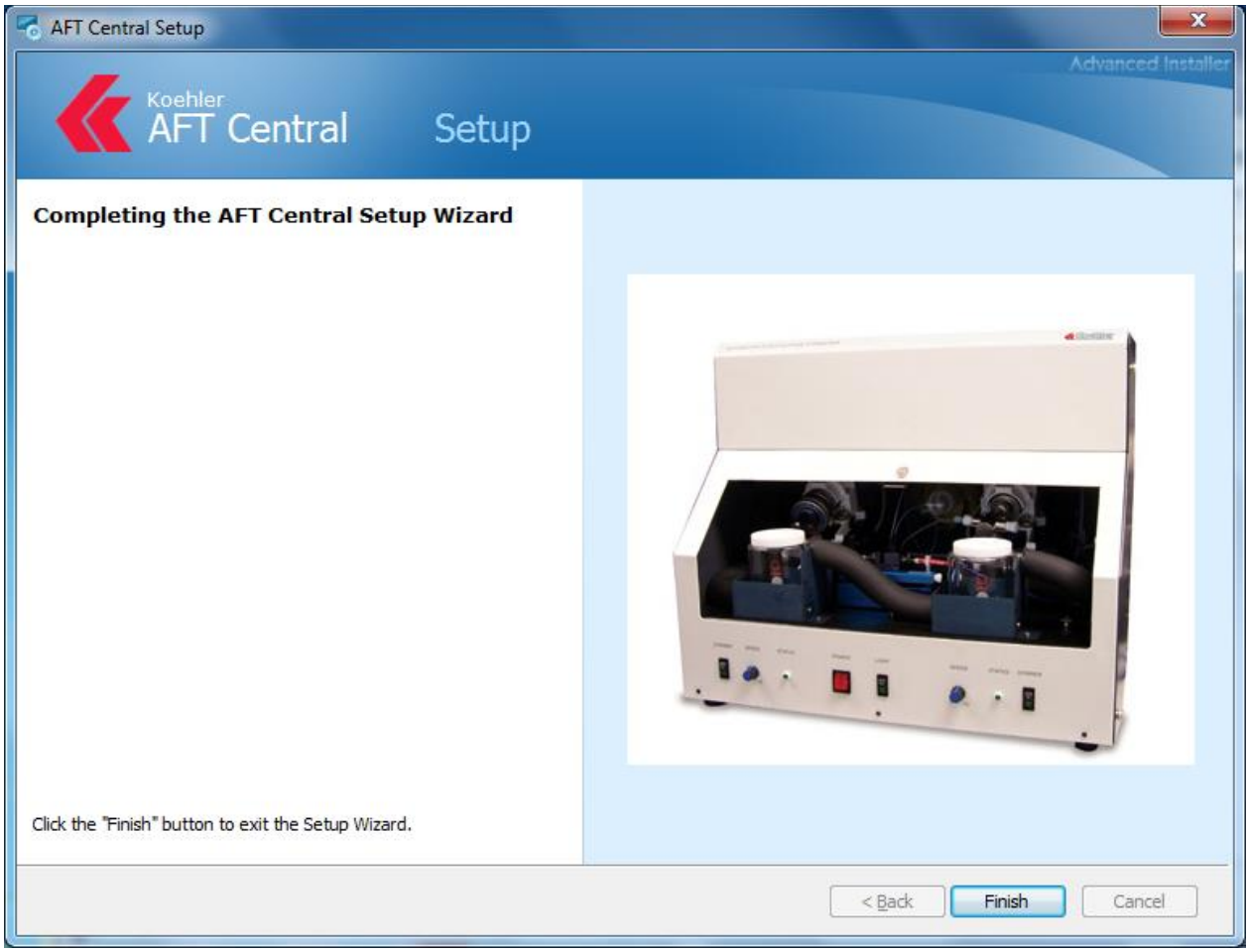
AFT Central Installation Screen. Click "Next" to continue



Click "Install" to begin AFT Central Installation.



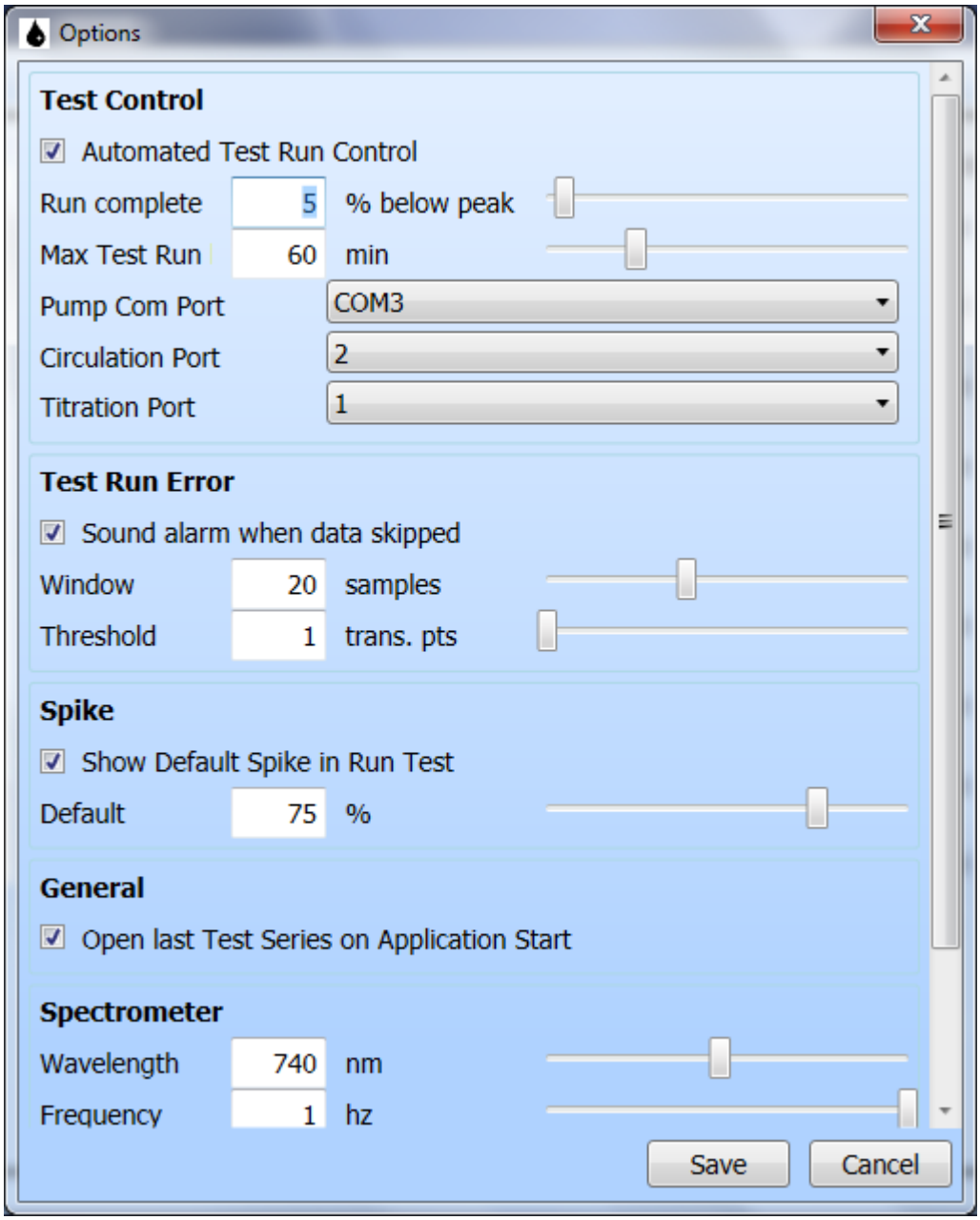
AFT Central installation in progress.



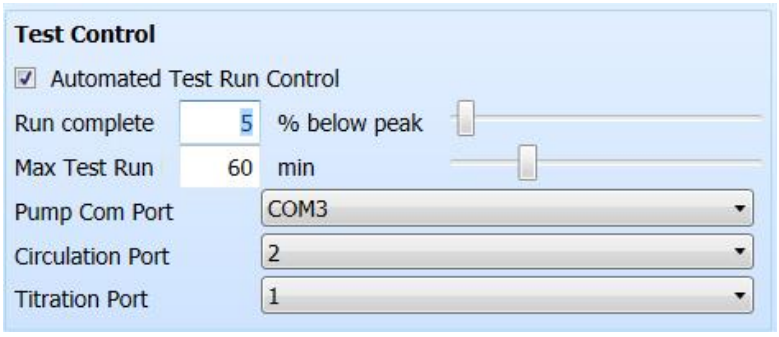
AFT Central installation is complete. Click "Finish" to exit the Setup Wizard.

3 Configuring AFT Central Options and Operation with OOIBase32 Platinum Edition

When opening the AFT Central software package for the first time, some options and settings may need to be configured. Open the AFT.exe shortcut icon. From the main screen toolbar select **Tools** then **Options**. The Options screen is pictured below:



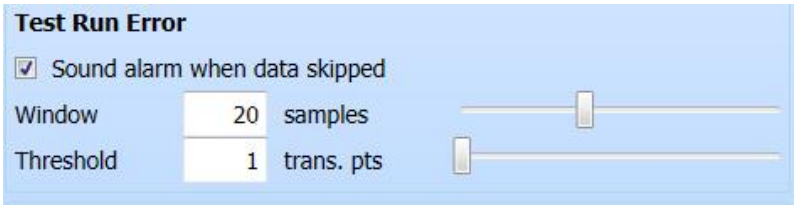
Several different options may be selected for specific applications. See Test Control section below:



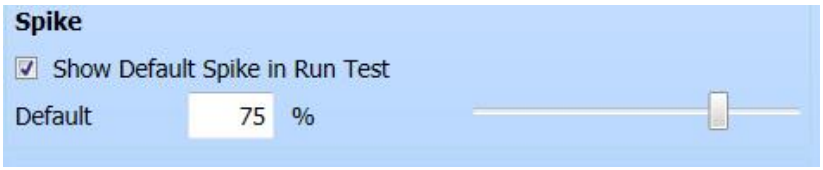
General options include Test Run Control settings which allow for automatic stopping of an experimental titration based on the **"% below peak"** change in detection percent Transmittance past the flocculation peak, and a maximum duration that a titration will run.

The **Max Test Run** length setting is a safety limit for test time, in case something goes wrong you will not pump until a technician spots the issue. This setting insures that the titration will stop automatically if a flocculation peak is never detected for a particular sample.

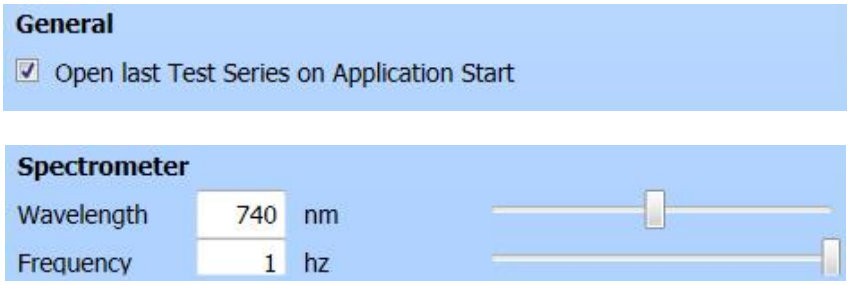
Finally, the **Pump Com Port** specifies the communication serial port connected to the pump drives. We do not use the blackbox anymore, we basically copied it and made our own. Pump Plug-in Port specifies the specific virtual COM port that the serial converter has been assigned. The pump ports are to ensure the user has the ability to operate both pumps as intended in case the communication is naturally switched.



The **"Test Run Error"** options (See Figure above) are used to filter out errant titration data point that may result in data spikes in a %T versus time plot, sometimes resulting from the development of air bubbles in the sample circulation pump system. An **alarm sound (literally it's a windows "ding" sound like the K71000 used to make)** may be activated to alert the operator that materials may be interfering with the titration. This may be useful in detecting clogs so then the operator can use the reverse pump function to save a test.



A titrant **Spike** volume option is also available with the present software. See Figure above. This option may be selected in order to add “spike” titrant to series of titration samples to cut down on testing time.(somehow with the setting as is, the spike was always 0 when I went to start the test, which I am okay with)



The options tab also features a General section and Spectrometer section. The General section gives the user the option to Open the last test series on Application Start. The Spectrometer section allows for the user to adjust the spectrometer emission settings. (like 3 settings got cut off from my screen shot)

4 Performing Titrations with AFT Central

6.1 Preparation of Test Samples

Test samples are prepared in 40 mL test tube reaction vials, and tests are performed by titrating a sample at three concentrations, 0.133 g/mL, to 0.267g/mL, for example. Prepare test samples by transferring 0.4000 g ± 0.0010 g, 0.6000 g ± 0.0010 g, and 0.8000 g ± 0.0010 gof heavy residua or asphalt to tared reaction vials and record the actual sample masses to an accuracy of ± 0.0005 g. For long-term storage of samples prior to testing, purge all samples with Argon (Ar) gas and seal vials with Teflon-lined caps. A day (16-24 hours) before testing, dissolve pre-weighed samples in 3.000 mL ± 0.001 mL of toluene (HPLC-grade). Re-seal reaction vials with Teflon-lined caps and store away from sunlight.

6.2 Calibrate the Circulation and Titrant Dispersion Pumps

To calibrate the titrant pump, clamp a 1.000 mL syringe-graduated cylinder to a small lab stand using a test tube clamp. Position the stand next to the titrant pump. With a stopwatch, time the flow rate of each titrant pump and adjust the flow rate to <0.500 mL/min (0.300 mL/min is recommended).

To calibrate the circulation pump, first check that the two metal knobs are tightened. Mount a 10.0 mL graduated cylinder to a small lab stand using a test tube clamp and position the stand next to a circulation pump. With a stopwatch, time the flow rate of each circulation pump and adjust the flow rate to 5.0 mL/min. To do this, first adjust course pump speed to speed setting “4” using the black knob with handle on right side of pump. Secondly, fine tune pump speed using the Speed knob on the front of the instrument.

Open AFT Central shortcut From the Main Screen (See Figure below), select **File**, then **New** to select a new Test Series.

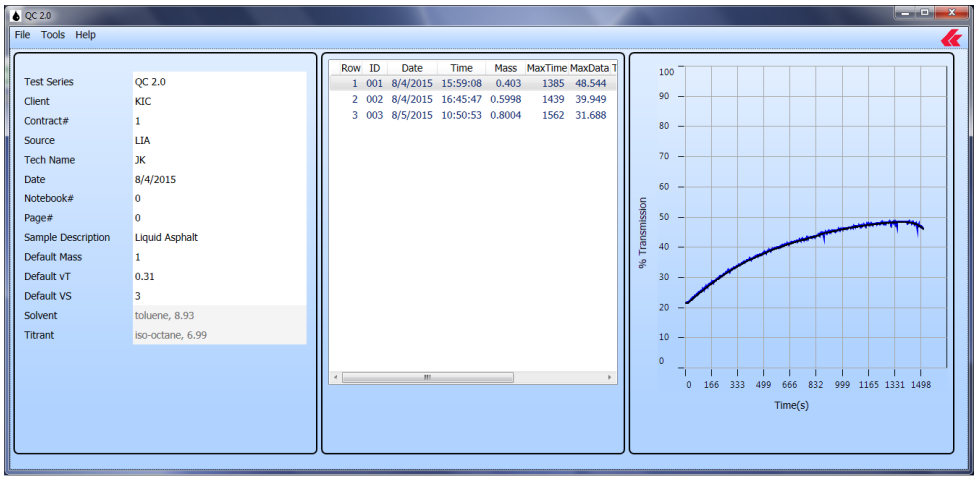
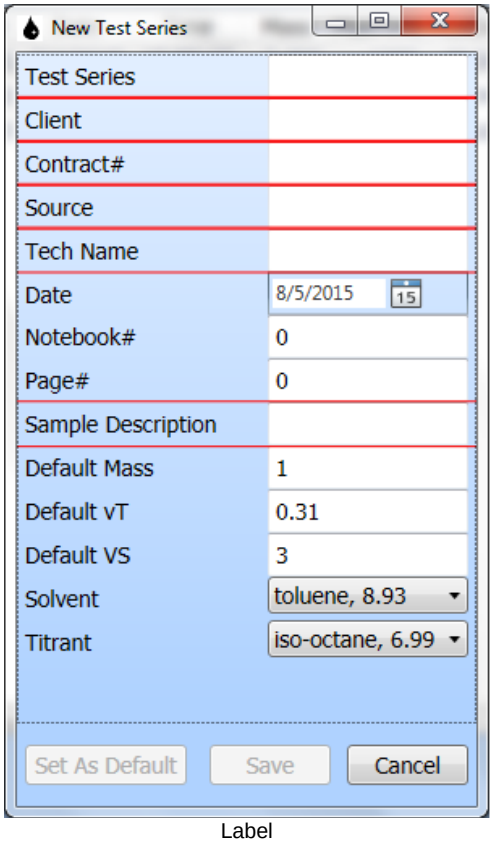


Figure label

The **New Test Series** window, (see Figure Below) is where you set up the parameters of a new test.

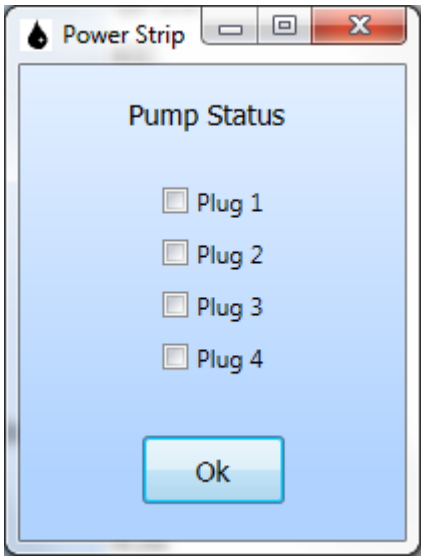


Label

NOTE: The software requires that all available input boxes contain information.

Once the Test Series input data values have been entered, click **Save** to exit the window.

Open the Power Strip panel by clicking **Tools** then **Power Strip** from the Main Screen toolbar. See Figure below:



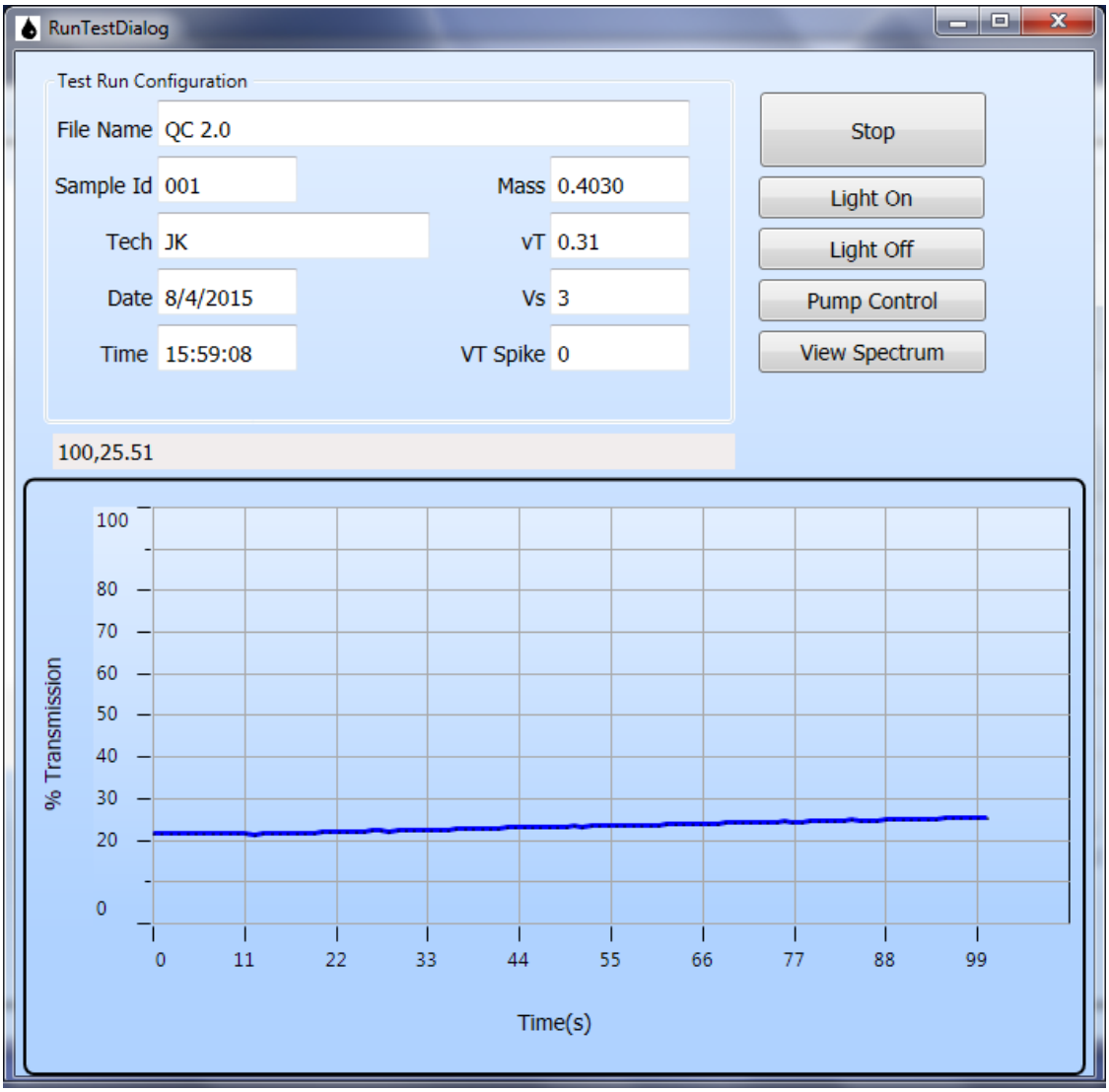
label

There is no wait time anymore. The Power Strip controls power to the Pumps. Plug 1 controls the Titrant (Left) Pump. Plug 2 controls the Circulation (Right) Pump.

6.3 To conduct a titration,

place a stir bar into a 40 mL test tube dude, whats with the suspicious quotes? vial containing a sample and screw a Teflon cap/reactor cover onto the reaction vial. Place the vial into a 250-mL jacketed reaction beaker and engage the stir bar and fill the jacketed beaker with water so that the sample inside the test vial is completely covered. Set the stir plate speed so that it creates a gentle vortex in the solution. Allow several minutes for the sample temperature to come to equilibrium, particularly if elevated temperature testing is being performed.

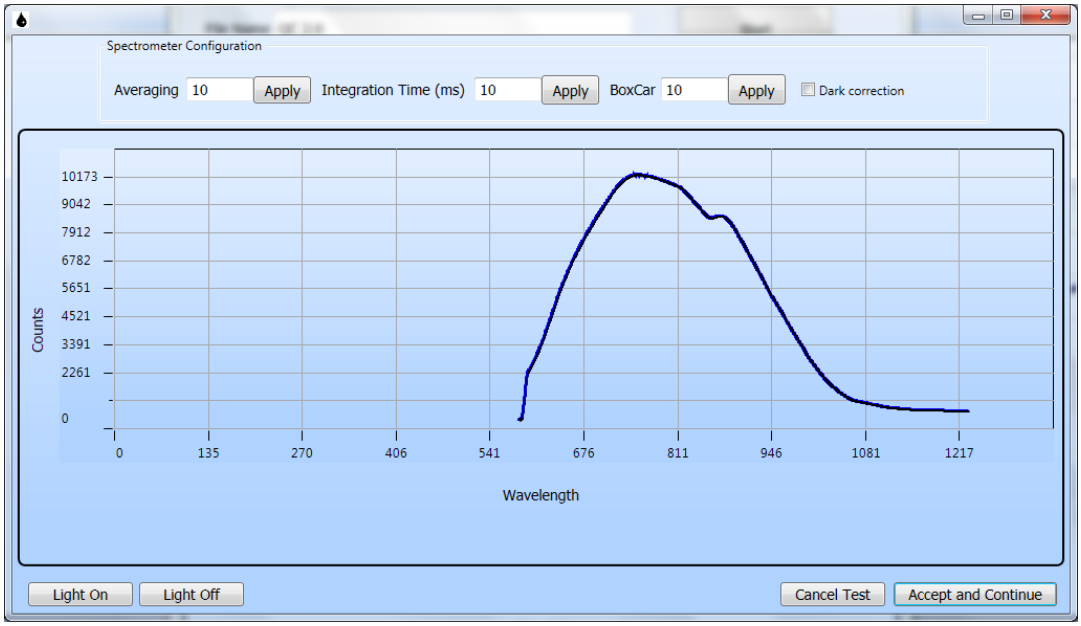
Select **Run Test** from the **Tools** pull-down menu on the Main Screen Toolbar. See Figure below:



Input a Sample ID, Mass of heavy residual or Asphalt,, and Volume of spike Titrant (0 if no Titrant is added during sample preparation) and hit start.

You will then be prompted that both pumps will begin circulating to purge. Make sure that the titration pump is recirculating iso-octance and the circulation pump is recirculating toluene.

The spectrometer screen will then appear to make sure the light source is working correctly. If you can see the spectrum as seen below, hit accept and continue.

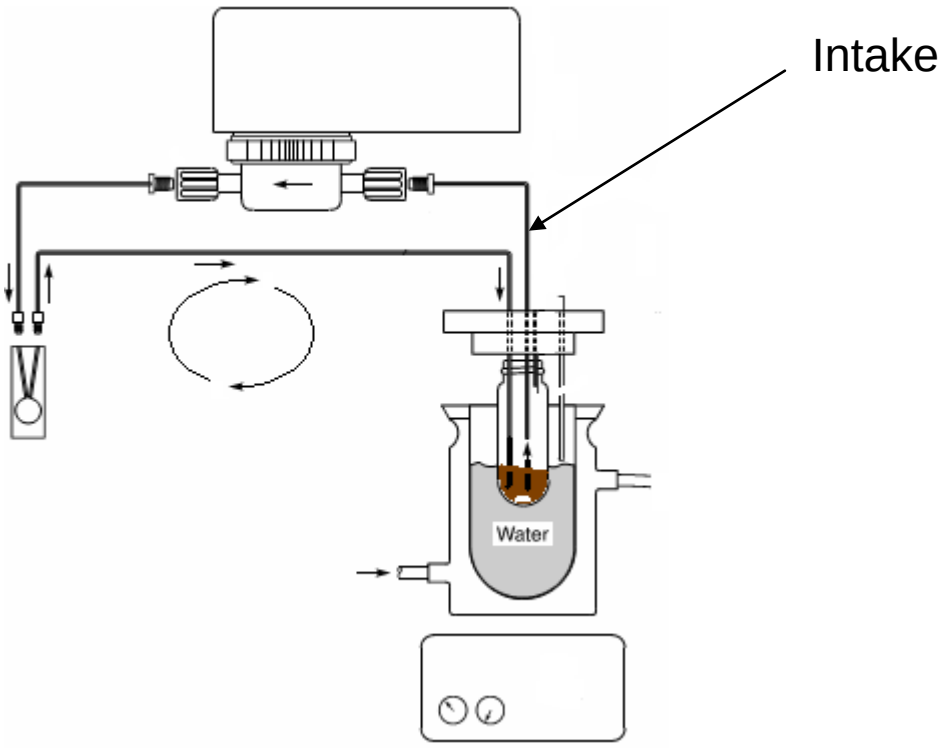


You will then be asked to zero the spectrometer. This will set a global light and dark for the test. You should hit continue when the circulation lines are full of pure, clean toluene to get an accurate light and dark.

Then the start test screen will appear. The titration pump will now shutoff and the circulation pump will continue to run. At this time you want to purge the toluene out of the lines by pumping air. After all the toluene is out, insert the tubes into the sample to start recirculating sample. When the spectrometer has stabilized, hit start test.

Once the titration test is complete and the instrument stops, select “OK” to accept the sample run data.

6.4 To clean the titration system, first pump the rest of the sample back into the vial by lifting the intake tube out of the sample and turning the pump on. Then remove the circulation pump intake tube completely and wipe clean with toluene before placing it into a small beaker containing toluene.



Sample circulation system; reaction vial, water-jacketed beaker, sample circulation pump, and flow cell

Remove the second tube and place it into the waste beaker pump toluene through the system until the waste toluene is clear. After a short period of time remove the tube from the waste beaker and re-circulate fresh toluene through the circulation loop flow cell. Remove the tested sample vial from the water jacket beaker and Teflon cap, dispose of the tested material and rinse the vial and air out the empty vial in a vented fume hood. Clean and dry the Teflon tubing cell and pump with a cold-trap vacuum line.

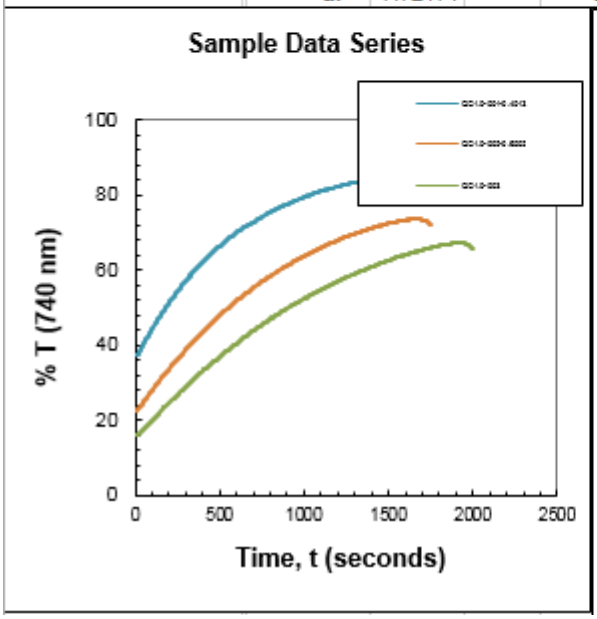
Proceed to test the remaining samples in the sample set as previously described.

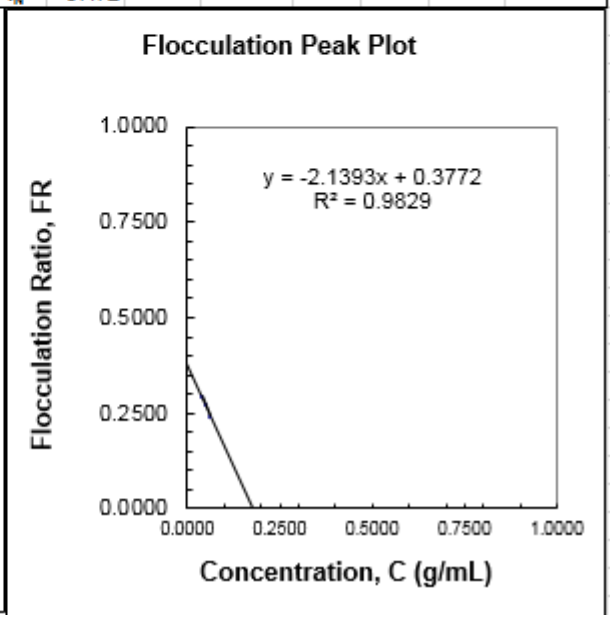
5 Analyzing the Data

When a set of titration data has been generated and accepted, select File then Export to Excel to generate a data spreadsheet. Save the spreadsheet with an appropriate name to a specified file. See Figure below:

	Flocculation Parameters										
Samples	v_T (ml/min)	T_o (s)	Wt (g)	V_s(ml)	V_t (ml)	C (g/m	FR	δ_{oil}			S_{oil}
QC1.0-001-0.4013	0.300	1455.1	0.4013	3.0000	7.2755	0.0391	0.2920	11.95	7.48	18.13	255.91
QC1.0-002-0.6002	0.300	1647.76	0.6002	3.0000	8.2388	0.0534	0.2669	11.73	5.00	13.73	244.14
QC1.0-003	0.300	1913.43	0.7996	3.0000	9.5671	0.0636	0.2387	11.94	3.75	11.96	255.32

Calculated	m =	-2.1393	p_a =	0.6228	m(V') =	1.67
Heithaus	y_0 =	0.3772	p_o =	2.5164	y_0(V') =	5.56
Parameters	C_{min} =	0.1763	P =	6.6718		
	dF =	7.72171	I_N =	37.72		





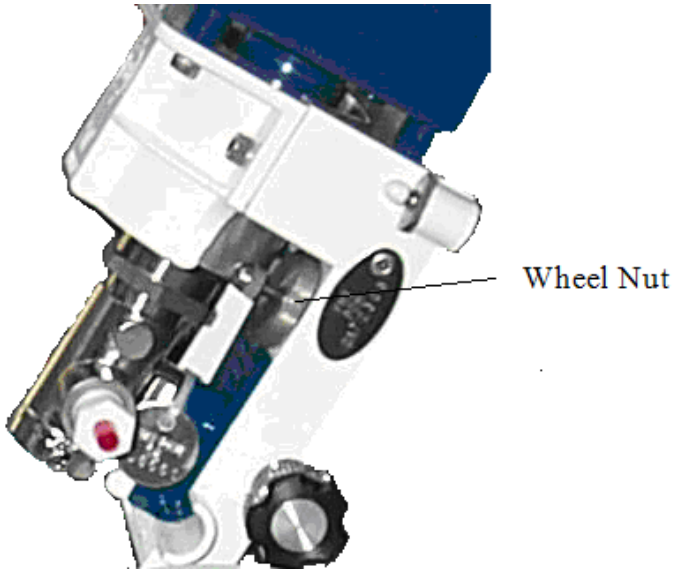
8 Maintenance and Troubleshooting

In order to keep the AFT instrument working properly, some routine maintenance is required, which will include periodic replacement of circulation pump seals and periodic cleaning of the quartz flow cell.

Routine Pump Seal Replacement:

It is strongly recommended that the two Rulon-fiber lip seals (red), located inside the circulation pump head, be replaced after every 100 hours that the instrument is in use.

8.1 To replace lip seals locate and unloosen completely the two wheel nuts, located on either side of the pump drove (Figures 1 and 2).

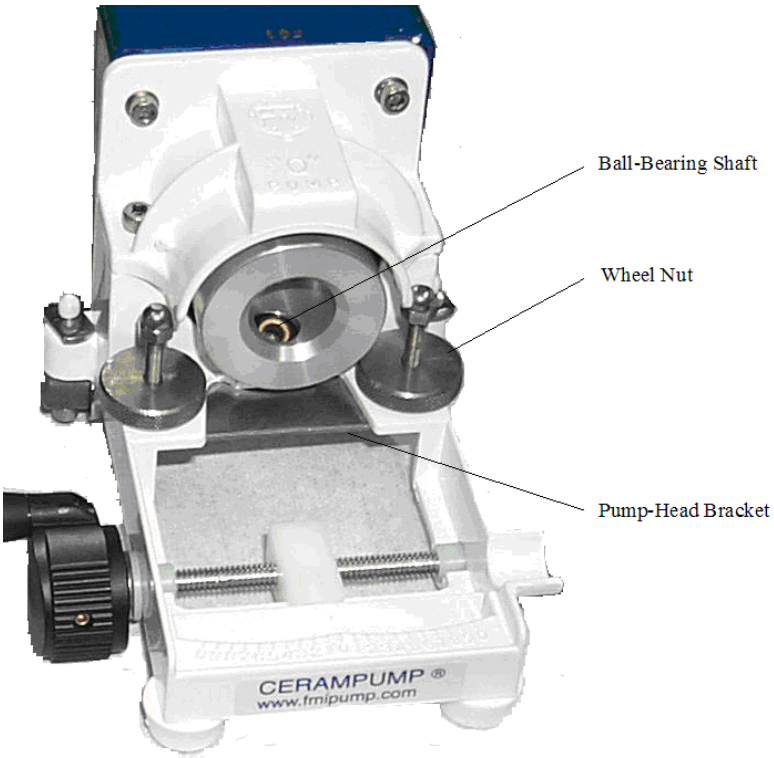


Pump head/drive assembly, *All Rights Reserved, Fluid Metering Inc., http://www.fmipump.com.*

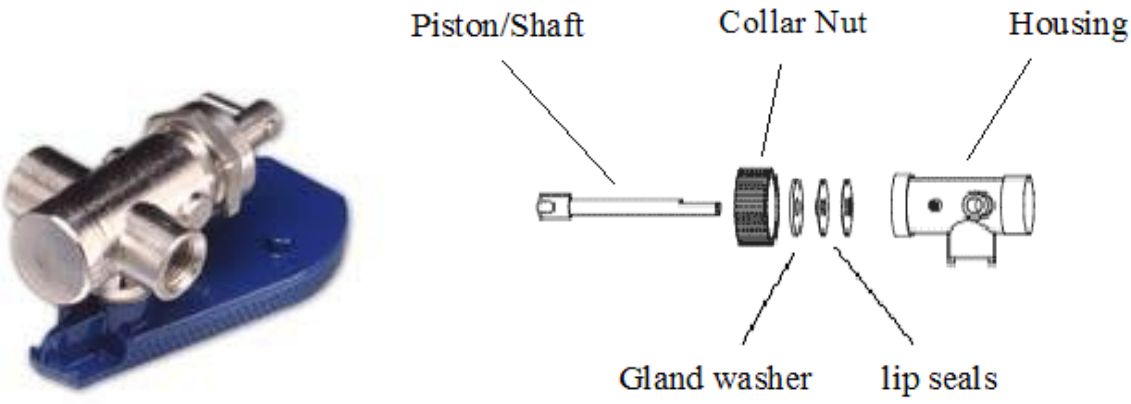
The pump head should move around loosely. Maneuver the pump head out and away from the bracket and pump drive noting how the pump head is notched into the drive bracket. Next, slide the metal piston pin, located at the back of the piston, out of the ball-barring shaft to completely remove the pump head.

With the pump head removed, completely loosen the collar nut to expose the gland washer and lip seals. Note how the holes in the lip seals are directionally positioned (beveled out). Slide the piston out of the housing and remove the old lip seals by sliding them off of the piston. Replace with new lip seals, to do so, while use a twisting action (i.e., gently twist the piston while sliding a lip seal onto the piston) using care not to tare the lip seal or “flip” the bevel. Slide the piston back into the housing, replace and tighten the collar nut, and reassemble the pump head to the drive. Re-assemble by sliding the

piston pin back into the ball-bearing shaft, then place the pump head stand (blue) into the pump drive bracket. NOTE: the pump head will need to be properly re-assembled so that the plastic catch which changes the direction of the pump head via the screw drive located toward the front of the pump drive fits with the pump head stand.



Pump drive, *All Rights Reserved, Fluid Metering Inc., http://www.fmipump.com.*.



Pump head, picture (left), schematic (right), *All Rights Reserved, Fluid Metering Inc., http://www.fmipump.com.*

A second important routine maintenance practice is to periodically inspect the quartz flow cell to assure that it is clean.

8.2 If the flow cell becomes clogged with particulate, try the following:

Prepare a concentrated solution of tri-sodium phosphate dissolved in distilled water. Pump this solution through the flow cell, periodically stopping the pump and allowing the solution to soak in the cell, then re-engaging the pump.

Quartz Flow Cell, *All Rights Reserved, Starna Cells Inc., http://www.starnacells.com.*

Under some circumstances it may be advantageous to change the flow direction of the pump during the “tri-sodium phosphate in water”. Use the crankshaft knob to reverse the direction of the pump by changing the angle of the pump head relative to the “0” mark located on the pump drive.

Once the flow has been cleaned, pump several 20 mL of acetone followed by several 20 mL of toluene through the pump to prime the pump.

9 Service

Under normal operating conditions and with routine maintenance, the Automatic Flocculation Titrimeter 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 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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Petroleum Testing & Analysis Instrumentation • Custom Design & Manufacturing

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