FULL AUTOMATIC ASPHALTENE ANALYZER

APD-500A

USER'S GUIDE

Please read this manual carefully to ensure correct use of this analyzer before you start to use it.

COSMO TRADE & SERVICE CO., LTD.

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1. Introduction

This user's guide explains you how to operate the APD-500A Full Automatic Asphaltene Analyzer, as a basic method.

2. Notices

This guide explains you about the APD-500A with its standard configuration.
For any specially added configuration, if any, please refer to a separate specification sheet.
Heating is needed when you prepare sample solution, so do your job in a draft chamber using an appropriate protective equipment in order to avoid to aspirate solution's vapor and protect from burn injury. It is requested that you read this user's guide carefully before you start to actually use this analyzer. Before operating this analyzer, its installation and setting of necessary parameters into the analyzer must have been carefully done.

2-1. Installation Requirement

1) Power supply

When about 30 minutes have elapsed after turning on the analyzer, select the "Function 8" to display "Potential" and make sure that there is no particular fluctuation between the right and left potential values (S and L in mV). If there is any fluctuation, check your local supply voltage and take a necessary precaution to install a voltage stabilizer, etc. when voltage fluctuation is taking place from time to time.

2) Installation space

- Make sure that a place of installation is flat.
- Make sure that your environment to use the analyzer is free from interfusion of dust, water or any other foreign object.
- Make sure that a place of installation is free from any vibration from its surrounding area. A light path will be misaligned due to vibration, and it may affect "Potential" (mV) and cause inaccurate measurements as a result.

3) Nozzle position and stirrer revolution

After setting a suction nozzle, put a magnetic stirring bar and n-Heptane in a flask. Set it on a turn-table and check suction and stirrer's revolution speed. If stirrer's revolution speed is too fast, air is sucked during the suction of sample solution, which will affect measurement values.

2-2. Handling of Standard Asphaltene Material (APD-006)

1) Heating a bottle of APD-006

Before taking standard Asphaltene material from a bottle, heat it as it is in a bottle in a thermostatic chamber kept at the temperature of 50 to 60°C for 30 minutes at minimum. Then, shake a bottle strongly so that its inner liquid may become uniform. If you take Asphaltene sample from a bottle without heating, Asphaltene particles are not uniform in the liquid, and using such a sample, it will be very difficult to get accurate results of measurements. Also, if Asphaltene liquid has cooled down during your sampling, accurate measurements will also become difficult due to the said reason, so in such a case, it will be needed to heat such once-cooled Asphaltene liquid again. Sampling as quickly as possible in a short time is therefore very important.

2) Protection from foreign object interfusion

When you open a bottle of APD-006, make sure first that there is no dust around you. Use a clean spatula or disposable pipette, preferably a new one every time when you take Asphaltene sample. Always close a bottle with a cap, except when you take Asphaltene sample from it, in order to protect from interfusion of foreign object.

3) Purging by nitrogen

After you used a bottle of APD-006, blow nitrogen gas into a bottle to exhaust oxygen inside. When oxygen remains inside a bottle, it may be possible that Asphaltene nature is transmuted.

4) Keeping a bottle of APD-006

Keep this bottle in a dark and cool place free from the sunlight. Light shielding is important as Asphaltene has a nature to deteriorate in quality by the sunlight.

2-3. ”0-100%” Calibration

This calibration must be done to make actual measurements correctly. It is needed for the purpose to compensate deviations of “Potential” that may result from deterioration in a light source lamp’s illumination intensity caused by supply voltage’s fluctuation, variation of surrounding temperature and humidity, and aged deterioration, as well as contamination of a flow cell (used for absorbance measurement), clouding of a translucent mirror and sensitivity deterioration of detectors, etc.

3. Primary Preparation of Apparatus, Reagents and Materials

3-1. Equipment, Reagents and Materials

3-1-1. Equipment

- Thermostatic chamber (50 to 60°C)
- Water bath (80°C)

3-1-2. Reagents and Materials

- Standard Asphaltene material (APD-006)
- Actual samples for measurements
- n-Heptane (reagent-grade, >99% purity)

3-2. Standard Asphaltene Material for Calibration and Actual Samples for Measurement

Heat bottles of APD-006 and actual samples for measurement in a thermostatic chamber that is kept at the temperature of 50 to 60°C for about 30 minutes to 1 hour.

3-3. Power Supply

Turn on the analyzer and make sure that its display indicates “READY”, and select “Potential” by “FUNCTION 8” to indicate the right and left values (S and L in mV) on the display. Leave the analyzer as it is for at least 20 minutes and make sure that there is no significant fluctuation of those values.

- **Confirmation of Potential**

The light emitted from a light source lamp passes through a flow cell and is separated by a translucent mirror to 2 wavelengths of S (750nm) and L (800nm), which are detected by 2 detectors as electric signals. The mV values of those electric signals are “Potential”.

Supposing that a flow cell is not contaminated and is empty, select “Potential” by “Function 8” to indicate the mV values on the display. If your analyzer is normal, both of the S and L values should be more than 800mV and stable without fluctuation. It is very important.

In case there is light source lamp’s deterioration in quality, the mV values may become extremely low, and in such a case, a lamp must be replaced with a new one.

This confirmation of potential must be done when more than half an hour has elapsed after you turned on the analyzer, and it is why you will have to wait for a light source lamp to become stable.

3-4. Heating n-Heptane

Heat a bottle of n-Heptane in a water bath kept at the temperature of 80ºC for about 30 minutes to 1 hour.

4. Preparation of Standard Solution and Sample Solutions

4-1. Apparatus, Reagents and Materials

4-1-1. Apparatus and labwares

- Analytical balance (with a capacity to weigh up to 0.1mg in minimum unit)
- Disposable pipette (around 1mL)
- Measuring pipette (measurable up to 1mL) or one-mark pipette (1mL)
- Measuring cylinder (100mL)
- Measuring flask with a stopper (500mL)
- Erlenmeyer flask with a stopper (100mL)
- Magnetic stirring bar

4-1-2. Reagents and Materials

- Toluene (reagent-grade, >99.5% purity)
- Heated n-Heptane (reagent-grade, >99% purity, see **“3-4. Heating n-Heptane”**)
- Standard Asphaltene material for calibration (APD-006) and actual samples for measurement

4-2. Standard Asphaltene Solution for Calibration

This is used to set the absorbances stated on a label of standard Asphaltene’s bottle (1g of Asphaltene in 100mL of Toluene; for 10mm cell) to your analyzer.

The “ABS(S):1gr/100ml Toluene and ABS(L):1gr/100ml Toluene” stated on a label of standard Asphaltene’s bottle indicate the absorbances for 10mm flow cell, but by doing this calibration, conversion to the analyzer’s 3mm cell is automatically calculated and saved into the analyzer’s internal memory as proper parameters.

4-2-1. Preparation of Standard Asphaltene Solution

1) Using an analytical balance, precisely weigh and take 10g of standard Asphaltene material (APD-006) that has been heated in a thermostatic chamber, and put it into a 500mL measuring flask.
2) Take 500mL of Toluene in the measuring flask and tightly put a stopper on it. Then, shake the flask strongly to dissolve and mix up the standard Asphaltene sample.

4-3. Making a Working Curve

All of your operation to prepare a standard Asphaltene solution till the end of measurement must be finished within 2 hours. Do not make any measurement using a standard Asphaltene solution that has been prepared more than 2 hours ago. It is why after 2 hours, Asphaltene particles in a solution will start agglomeration and particle size distribution will vary, with a result of inaccurate measurements.

Using this analyzer, a working curve can be made by measuring 5 points.
Weigh and take standard Asphaltene material by 0.1g, 0.3g, 0.5g, 0.7g and 0.9g, and add 1mL of Toluene and 100mL of heated n-Heptane to each standard sample for dissolution. After the prepared standard Asphaltene solutions have cooled down to a room temperature, you can measure Asphaltene particles that are separated out.
From “ASTM Value mass%” stated on a label of standard Asphaltene material’s bottle, it is possible to calculate Asphaltene amount using the formula “Weighed Asphaltene sample’s amount (g) x ASTM Value mass%”.

4-3-1. Preparation of Standard Asphaltene Solution

1) Using an analytical balance, weigh and take pre-heated 5 standard Asphaltene samples (APD-006), and put them into 5 pieces of Erlenmeyer flask of 100mL each with stopper.
2) Take and add 1mL of Toluene each using a measuring pipette (1mL measurable) or a one-mark pipette (1mL).
3) Using a water bath, by which n-Heptane is being heated, heat the said Erlenmeyer flasks with stoppers, shaking them slowly and horizontally from time to time, until standard Asphaltene samples precipitated on the bottoms will be entirely dissolved.

<Note: Complete dissolution by Toluene>
While heating Erlenmeyer flasks with standard Asphaltene samples by a water bath (or a hot plate) as mentioned above, look through by illumination a bottom of each flask and make sure that there is no more Asphaltene sample stuck and remaining on a bottom.
When heating Erlenmeyer flasks, loosely put stoppers. If stoppers are tightly put, they may be blown due to internal Toluene’s vaporized pressure.

4) After you confirmed complete dissolution of Asphaltene sample by Toluene, add 100mL of n-Heptane heated up to 80°C to each Erlenmeyer flask. At this time, a measuring cylinder used to take 100mL of n-Heptane should have been also heated to the same temperature as that of n-Heptane. If this cylinder is cool, a temperature of n-Heptane will also come down and it will affect results of measurements.
When you have added heated n-Heptane, tightly put a stopper on a flask and shake it strongly up and down and right and left. Continue its shaking until a flask’s temperature comes down to a room temperature at intervals of about 5 to 10 minutes. A stopper should be loosened while a flask is left cooling. If it is tightened, there will be a possibility that a stopper cannot be taken off later due to lowering of internal pressure.

<Note: Time for cooing>
Although it depends on a surrounding condition, it usually takes about 40 minutes from adding heated n-Heptane until a flask’s temperature comes down to a room temperature. Do its cooling in a room where the temperature is kept at 20 to 30°C. If an ambient temperature is extremely high or low, or if a variation in temperature is so serious, particle size distribution and

amount of Asphaltene particles to be separated out will vary and cause an error of measurement values.
It is reminded that you continue to shake a flask with standard Asphaltene solution at intervals of 5 to 10 minutes until a flask's temperature comes down to the room temperature.

Furthermore, all of such your operation to prepare a standard Asphaltene solution till the end of measurement must be finished within 2 hours. It will not be allowed for you to make a measurement using a standard Asphaltene solution that has been prepared more than 2 hours ago. It is why after 2 hours, Asphaltene particles in a solution will start agglomeration and particle size distribution will vary, with a result of inaccurate measurement.

4.4. Sample Solutions for Actual Measurement

Preparation of a sample solution for actual measurement will be done in the same way as mentioned above for making a working curve.
Amount of a sample to weigh and take should be determined so as to come within the range of a working curve's concentration that was made by standard Asphaltene solution prepared to make a working curve.

4.4.1. Determination to Weigh and Sampling

Refer to the following table regarding a weight of a sample to take.

Asphaltene content (wt%)	Sampling weight (g)	Weighing accuracy (g)
1.0 or less	3 ± 0.3	0.01
Over 1.0 to less than 3.0	1 ± 0.1	0.005
Over 3.0 to less than 10	0.3 ± 0.03	0.001
Over 10 to less than 15	0.1 ± 0.01	0.0005

Determine a sample's weight so that it may come within the range of Asphaltene content measured using a working curve. For a measurement of an unknown sample, weigh and take 0.3g to measure, and from its measured value, recalculate a weight to take so that it comes within the range of Asphaltene content measured using a working curve. Then, prepare a proper sample solution and make your measurement. As a rare case, due to extraordinary large Asphaltene content, there will be a case that a measured value does not come within the range of Asphaltene content measured using a working curve despite you measured a sample that was weighed according to your recalculated weight. In such a case, from its measured value, recalculate again a weight to take as mentioned above, prepare a sample solution and make your measurement again.

4.4.2. Confirmation of "A=ABS S/ABS L" value

There will be a case that its value does not come within the range of 1.0 to 1.3 due to your analyzer's disorder or a light source lamp's deterioration in quality, etc. So, confirm this value whenever you make your measurement.

5. Procedure to Operate APD-500A

5-1. 0-100% Calibration

1) Press “FUNC”, ”0” and ENTER.
2) Select “Calibration ON” and press ENTER.
3) Select “Calib 1 for APD” and press ENTER.
4) Select “Set 0%T” and press ENTER: A shutter for a light source lamp closes.
5) Values of S and L in mV will be indicated on the display, so press ENTER when they have become stable around 0.
6) Select again “Set 100%T” and press ENTER: A shutter for a light source lamp opens.
7) Set a flask of 500mL n-Heptane at a suction nozzle, and press ”Samp”. A suction pump starts running and n-Heptane is sucked.
8) Values of S and L in mV will be indicated on the display, and press ENTER when they have become stable.
9) Press ”Samp”.: A suction pump stops, and suction of n-Heptane stops.
10) Press “RESET” and make sure that ”READY” is indicated on the display.

5-2. Cell Length and Wavelength Calibration

5-2-1. Input of 750nm and 800nm Absorbance Values

1) Press “FUNC”, ”0” and ENTER.
2) Select “Calibration OFF” and press ENTER.
3) Select “S1 of STD-1 (n-Heptane)”: 750nm absorbance “0” and press ENTER.
4) Select “L1 of STD-1 (n-Heptane)”: 800nm absorbance “0” and press ENTER.
5) Select “S2 of STD-2 (Standard Asphaltene solution for calibration)”: Input 750nm absorbance “**S2**” and press ENTER.

   S2= Weight of standard Asphaltene sample (APD-006) contained in 100mL of standard Asphaltene solution for calibration (Toluene) x 750nm absorbance (ABS(S): 1gr/100ml Toluene) stated on a label of standard Asphaltene material’s bottle

   Weight of standard Asphaltene sample (APD-006) contained in 100mL of standard Asphaltene solution for calibration (Toluene) means **“Precisely weighed 10g of standard sample (APD-006) ÷ 5”** when 10g of standard sample (APD-006) was dissolved by 500mL of Toluene in a 500mL measuring flask.
6) Select “L2 of STD-2 (Standard Asphaltene solution for calibration)”: Input 800nm absorbance “**L2**” and press ENTER.

   L2= Weight of standard Asphaltene sample (APD-006) contained in 100mL of standard Asphaltene solution for calibration (Toluene) x 800nm absorbance (ABS(L): 1gr/100ml Toluene) stated on a label of standard Asphaltene material’s bottle

   Weight of standard Asphaltene sample (APD-006) contained in 100mL of standard Asphaltene solution for calibration (Toluene) means **“Precisely weighed 10g of standard sample (APD-006) ÷ 5”** when 10g of standard sample (APD-006) was dissolved by 500mL of Toluene in a 500mL measuring flask.
7) Continue to press ENTER until ”READY” is indicated on the display.

5-2-2. Wavelength Calibration

1) Press “FUNC”, ”0” and ENTER.
2) Select “Calibration ON” and press ENTER.
3) Select “Calib 2 for APD” and press ENTER.
4) Select “Set STD-1 Soln !” and press ENTER.

5) Set a flask of 500ml n-Heptane at a suction nozzle, and press “Samp”. A pump starts running and n-Heptane is sucked.
6) S and L values in mV will be indicated on the display, and press ENTER when those values have become stable at around “0.00” in about 10 to 20 seconds.
7) Press ”Samp”. A pump stops running and suction of n-Heptane stops.
8) Select “Set STD-2 Soln !” and press ENTER.
9) Set a flask of standard Asphaltene solution for calibration at a suction nozzle, and press ”Samp”. A pump starts running and standard Asphaltene solution is sucked.
10) S and L values in mV will be indicated on the display, and press ENTER when those values have become stable in about 10 to 20 seconds.
11) Press ”Samp”. A pump stops running and suction of standard Asphaltene solution stops.
12) Press “RESET” and make sure that ”READY” is indicated on the display.

6. Procedure to Make a Working Curve

6-1. Input of Values

1) Press “FUNC”, ”0” and ENTER.
2) Select “Calibration OFF” and press ENTER.
3) Press ENTER three times to indicate “WT% *.**”.
4) Input the value of ”ASTM VALUE *.** mass%” stated on a label of standard Asphaltene material’s bottle, and press ENTER.
5) Input the value of ”M.abs = *.**” stated on a label of standard Asphaltene material’s bottle, and press ENTER.
6) Input the value of “CAL-1 *.****” (accurate weight of sample used to make a 0.9g working curve) and press ENTER.
7) Input the value of “CAL-2 *.****” (accurate weight of sample used to make a 0.7g working curve) and press ENTER.
8) Input the value of “CAL-3 *.****” (accurate weight of sample used to make a 0.5g working curve) and press ENTER.
9) Input the value of “CAL-4 *.****” (accurate weight of sample used to make a 0.3g working curve) and press ENTER.
10) Input the value of “CAL-5 *.****” (accurate weight of sample used to make a 0.1g working curve) and press ENTER.
11) Continue to press ENTER until “Function (0-9) ?” is indicated on the display.
12) Press “RESET” and make sure that ”READY” is indicated on the display.

6-2. Confirmation or Change of Measuring Parameters

1) Press “FUNC”, ”1” and ENTER.
2) Set “Waiting Time” to “0” and press ENTER.
3) Set “Pump 1 Running Time” to “10” (seconds) and press ENTER.
4) Set “Pump 2 Running Time” to “10” (seconds) and press ENTER.
5) Set “Washing Time” to “10” (seconds) and press ENTER.
6) Select “CHG Seq 1-3 ?” and ”3”, and press ENTER.
   (1: Without washing, 2: With washing, **3: Make a working curve)**
7) Press “RESET” and make sure that ”READY” is indicated on the display.

6-3. Input of Calculation Coefficient File

1) Press “FUNC”, ”2” and ENTER.
2) Select “File No. ?” and ”1”, and press ENTER.
3) Continue to press ENTER until “Function (0-9) ?” is indicated on the display.
4) Press “RESET” and make sure that ”READY” is indicated on the display.

6-4. Measurements of Standard Asphaltene Solution to Make a Working Curve

1) Set a flask of 500mL n-Heptane at a suction nozzle, and press ”Samp”. A pump starts running and n-Heptane is sucked.
2) When a suction line and a pump are filled by n-Heptane, press ”Samp”.
3) Set on an auto-changer’s turn-table flasks of 0.9g to 0.1g standard Asphaltene solutions from the dense to thin solutions in this turn. Do not forget to put a magnetic stirring bar into each flask.
4) Press “START” to start measurements.
5) When measurements are over, set a flask of 500mL Toluene at a suction nozzle, and press ”Samp”. A pump starts running and Toluene is sucked.
6) When a flow line has been washed, press ”Samp”.
7) Set a flask of 500mL n-Heptane at a suction nozzle, and press ”Samp”. A pump starts running and n-Heptane is sucked.
8) When a flow line has been washed, press ”Samp”.

6-5. Coefficient Calculation and Saving Calculation Result’s File

1) Press “FUNC”, ”0” and ENTER.
2) Select “Calibration ON” and press ENTER.
3) Select “Calibration 3 for JPI” and press ENTER.
4) Select “Calib? ON” and press ENTER.

<Note>

By the above operation, the analyzer’s internal factor calculation will be done and its result will be saved into a built-in memory. Without doing this operation, there is no saving of factor calculation result into a memory.

<Confirmation of “A=ABS S/ABS L” value>

Its value has been adjusted to come into the range of 1.0 to 1.3 at the time of shipment of the analyzer. Confirm its value when you make measurements as there may be a case that it does not come into the said range due to a disorder of the analyzer or a light source lamp’s deterioration in quality. Results of measurements to make a working curve will be printed as ”Calibration Values” by pressing “FUNC”, “0” and “PRT” in this order.

6-6. Change of Measuring Parameters

1) Press “FUNC”, ”1” and ENTER.
2) Set “Waiting Time” to “0” and press ENTER.
3) Set “Pump 1 Running Time” to “10” (seconds) and press ENTER.
4) Set “Pump 2 Running Time” to “8” (seconds) and press ENTER.
5) Set “Washing Time” to “10” (seconds) and press ENTER.
6) Select “CHG Seq 1-3 ?” and ”1”, and press ENTER.
   (**1: Without washing**, 2: With washing, 3: To make a working curve)
7) Press “RESET” and make sure that ”READY” is indicated on the display.

7. Measurements of Actual Sample Solutions

1) Set on an auto-changer's turn-table flasks of actual sample solutions including magnetic stirring bars from No.1 position.
2) After the last flask of actual sample solution, set a flask of pure Toluene and another flask of pure n-Heptane.
3) Press "SIZE".
4) For the turn-table's start number, total sample numbers (actual sample solutions + 2), each sample number (ID), each sample weight (SW(g)), Toluene and n-Heptane, input "0" together with each ID and SW.
5) Press "START".
6) If you have set a larger number than the turn-table's maximum capacity, rotation of turn-table stops at the last position. In this case, set again other flasks of actual sample solutions on the turn-table and press "START" to continue measurements.

8. Trouble-shooting

Symptom	Possible cause	Countermeasure
No display indication even if you turned on the power.	• A power cord is not plugged to a wall socket. • A fuse has blown off.	• Check a power cord. • Replace a fuse with a new one.
Potential of a light source becomes less than 800.	• Deterioration due to a lamp's lifetime • Contamination of a flow cell (used to measure absorbance)	• Replace a lamp with a new one. • Clean the cell or replace it with a new one.
Working curve cannot be made nicely.	• Voltage of your power source is not stable.	• Secure a stability of your power source (voltage is liable to fluctuate if other pumps and heaters are connected to the same line of power source.)
In particular, 5 points of S/L values of your samples to make a working curve do not come into the range of 1.0 to 1.3. Measured values of your actual samples are not stable.	• Contamination of a flow cell • Deterioration of a lamp • Suction nozzle is not in the right position. • Suction by pump is not in good order. • Instability of potential values due to your touch on an optical mirror.	• Clean the cell or replace it with a new one. • Replace a lamp with a new one. • Check the position of a suction nozzle and tightening of joints. • Replace a pump with a new one. • Don't touch a mirror at easy option before the above operation.
Factor does not change even if a working curve was made.	• After making a working curve, you have not made factor calculation and memory saving.	• After making a working curve, select "Function 0" immediately, and making "Calibration ON", set "Calib JPI?" ON at "Calibration 3 for JPI".

Air bubbles come into a flow line from a suction nozzle.	• Air is sucked from a sample suction line. • Revolution speed of a magnetic stirrer is too fast.	• Check the position of a suction nozzle and tightening of joints. • Adjust the revolution speed at your measurement.
Once you measured high concentration samples, measurement values of next samples show higher values.	• Sample suction is not nicely done, and previous sample still remains in the cell. • Due to contamination of a flow cell, sample for measurement is not replaced with a next one in good order. • Due to disorder of pump's suction, sample's flow has become bad.	• Check the position of a suction nozzle and tightening of joints. • Clean the cell or replace it with a new one. • Replace a pump with a new one.
You hear abnormal noise from a pump.	• Deterioration of a pump	• Replace a pump with a new one.

1) Replacement of a light source lamp

When a light source lamp has been confirmed to be deteriorated in quality through your "Potential" confirmation, replace a lamp with a new one after making sure that a lamp has cooled down enough.

This lamp is a hand-made one, so a filament's position will be slightly different lamp by lamp, and by this reason, there will be a case that a value of "Potential" will not go up. In such a case, adjust a position of a lamp fixing screw or rotate a lamp fixing position for 90º so that you can get 800mV or more "Potential" value.

Never touch a lamp with your bare hands. If your hand's sebum attaches to a lamp, it will get clouded, and cause to lower illumination intensity.

2) Adjustment of a translucent mirror

If a value of "Potential" does not go up even after your replacement of a lamp, or in case the values of S (750nm) and L (800nm) are extremely different, there will be a possibility that a translucent mirror's position or its deterioration in quality is a cause of such problems.

Except the case that a mirror's fixing screw has explicitly loosened, you will have to note that a light path will be misaligned if you touch a mirror's position, and it will finally require a special adjustment to be professionally done by returning the analyzer to our factory.

9. Quick Reference for Operation Manual

9-1. Primary Preparation

1) Put bottles of standard Asphaltene material and actual sample for measurement in a thermostatic chamber kept at the temperature of 50 to 60ºC. : 30 min. to 1 hour

2) Turn on the analyzer for warming up. READY : 20 min. or more

3) Put a flask of n-Heptane in a water bath kept at the temperature of 80ºC. : 30 min. to 1 hour

9-2. Preparation of Standard Asphaltene Solution for Calibration and Sample Solution for Actual Measurements

500mL n-Heptane	: for 0-100% calibration
500mL measuring flask	: to make standard Asphaltene solution for calibration
100mL Erlenmeyer flask with stopper	: x 5 pieces
R (n-Heptane)	: 100mL Erlenmeyer flask with stopper
S (Toluene)	: 100mL Erlenmeyer flask with stopper

1) Weigh and take heated standard Asphaltene material for calibration in a 500mL measuring flask.	: 10g : Use a new pipette whenever you weigh and take a sample. : Put a glass stopper to Erlenmeyer flask while weighing.

2) Weigh and take heated actual sample (or standard Asphaltene material for calibration) in a 100mL Erlenmeyer flask. *** Asphaltene content in standard solution to make a working curve: ① 0.035g, ② 0.028g, ③ 0.020g, ④ 0.012g and ⑤ 0.005g/ 100mL**	: 0.1g, 0.3g, 0.5g, 0.7g and 0.9g (standard Asphaltene solution to make a working curve) : Use a new pipette whenever you weigh and take a sample. : Put a glass stopper to Erlenmeyer flask while weighing.

3) **Preparation of standard Asphalatene solution for calibration (Continued from 1) above):**
Take Toluene by 500mL using a measuring cylinder, and put it into a 500mL measuring flask with 10g standard Asphaltene material for calibration. Then, shake it to mix well.

4) **Preparation of sample solution for actual measurements or standard Asphaltene solution to make a working curve (Continued from 2) above):**

a. Using a pipette, take Toluene by 1mL in each 100mL flask, put it in a water bath to heat and shake it to mix well from time to time.

b. Take heated n-Heptane by 100mL using a 100mL measuring cylinder, put it into each 100mL flask and shake it to mix well. : A measuring cylinder should have been heated beforehand.

c. Put a magnetic stirring bar into each 100mL flask.

d. Cool down each 100mL flask with sample solution to the room temperature for 40 minutes. : All the measurements must be done and finished within 2 hours.

9-3. Preparation of the Analyzer

9-3-1 “0-100%” Calibration

(1)	F	0	ENTER	: Select Function No.0.
(2)	-	ENTER		: Calibration ON
(3)	-	ENTER		: Calib 1 for APD
(4)	ENTER			: Set 0%T (A shutter for a light source lamp closes.)
(5)	ENTER			: Set 0%
(6)	ENTER			: Set 100%T (A shutter for a light source lamp opens.)
(7)	Set a flask of 500mL n-Heptane at a suction nozzle.			
(8)	Samp			: Suction pump starts running.
(9)	ENTER			: Values of S & L will become stable in about 10-20 sec.
(10)	ENTER			: Set S & L values.
(11)	Samp			: Suction pump stops running.
(12)	RESET			: “RESET” will be indicated on the display.
(13)	Take off a flask of 500mL n-Heptane from a suction nozzle.			
(14)	Samp			: Suction pump starts running.
(15)	Samp			: Suction pump stops running at 10 seconds later.

9-3-2 Cell Length and Wavelength Calibration

1) Input of 750nm and 800nm absorbance values

(1)	F	0	ENTER	: Select Function No.0.
(2)	ENTER			: Calibration OFF
(3)	0	ENTER		: S1 of STD-1 (n-Heptane): 750nm absorbance “0”
(4)	0	ENTER		: L1 of STD-1 (n-Heptane): 800nm absorbance “0”
(5)	***.****	ENTER		: S2 of STD-2 (Standard for calibration): 750nm absorbance “***.****”
(6)	***.****	ENTER		: L2 of STD-2 (Standard for calibration): 800nm absorbance “***.****”

S2: Weighed and taken standard Asphaltene material (g) x 750nm absorbance (as stated on a bottle’s label) ÷ 5

L2: Weighed and taken standard Asphaltene material (g) x 800nm absorbance (as stated on a bottle’s label) ÷ 5

(7)	ENTER	x 12 times		: Continue to press until “READY” is indicated on the display.
	:			

2) Cell Wavelength Calibration

(1)	F	0	ENTER	: Select Function No.0.
(2)	-	ENTER		: Calibration ON
(3)	-	ENTER		: Calib 2 for APD → Set STD-1 Soln !
(4)	Set a flask of 500mL n-Heptane at a suction nozzle.			
(5)	Samp			: Suction pump starts running.
(6)	ENTER			: Values become stable in about 10-20 sec. → “0.00 0.00”
(7)	ENTER			: Set values.
(8)	Samp			: Suction pump stops running.
(9)	Take off a flask of 500mL n-Heptane from a suction nozzle.			

(10) ENTER : “Set STD-2 Soln !” will be indicated on the display.

(11) Set a flask of standard Asphaltene solution for calibration at a suction nozzle.

(12) Samp : Suction pump starts running.

(13) ENTER : Values become stable in about 10-20 sec. → “*.*** *.***”

(14) ENTER : Set values.

(15) Samp : Suction pump stops running.

(16) Take off a flask of standard Asphaltene solution for calibration from a suction nozzle.

(17) RESET : “READY” is indicated on the display.

9-3-3 Making a Working Curve

1) Input of values

(1) F 0 ENTER : Select Function No.0.

(2) ENTER : Calibration OFF

(3) ENTER x 3 times : for confirmation only

:

(4) WT% *.** ENTER : Input standard Asphaltene material’s wt%.

(5) Mabs *.*** ENTER : Input soluble absorbance of standard Asphaltene material.

(6) CAL-1 *.**** ENTER : Input weight of standard Asphaltene sample 1 (dense one)

(7) CAL-2 *.**** ENTER : Input weight of standard Asphaltene sample 2

(8) CAL-3 *.**** ENTER : Input weight of standard Asphaltene sample 3

(9) CAL-4 *.**** ENTER : Input weight of standard Asphaltene sample 4

(10) CAL-5 *.**** ENTER : Input weight of standard Asphaltene sample 5 (thin one)

(11) ENTER x 10 times : for confirmation only

:

2) Confirmation or change of measuring parameters

(1) F 1 ENTER : Select Function No.1.

(2) 0 ENTER : Waiting time → 0 minute

(3) 10 ENTER : Pump 1 running time → 10 seconds

(4) 10 ENTER : Pump 2 running time → 10 seconds

(5) 10 ENTER : Washing time → 10 seconds

(6) CHG Seq 1-3 ? : 1 → Without washing

3 ENTER : 2 → With washing

: 3 → Make a working curve.

(7) Function (0-9) ? RESET : “READY” is indicated on the display.

3) Input of calculation coefficient file

(1) F 2 ENTER : Select Function No.2.

(2) 1 ENTER : Specify a file number.

(3) ENTER x 9 times : for confirmation only

(4) Function (0-9) ? RESET : “READY” is indicated on the display.

4) Measurement of standard Asphaltene solution for calibration

(1)	Samp	: Suction of n-Heptan starts. : Before measurements, fill a suction line and a
(2)	Samp	: Pump stops running pump with n-Heptane to avoid a suction error.

Set flasks of solutions on a turn-table from dense to thin ones in this order.

(3)	START	: Measurement starts. : Measure from dense to thin solutions.

After measurements, wash a flow line and a pump.

(4)	Samp	: Toluene is sucked and drained.
(5)		: n-Heptane is sucked and drained.
(6)	Samp	: Pump stops running

5) Coefficient calculation and saving a file of calculation results

(1)	F	0	ENTER	: Select Function No.0.
(2)	-	ENTER		: Calibration ON
	-			: Calibration 1 for APD → Calibration 2 for APD
	-	ENTER		: Calibration 2 for APD → Calibration 3 for JPI
	-	ENTER		: Calib JPI ? ON
(3)	Function (0-9) ?		RESET	: "READY" is indicated on the display.

6) Change of measuring parameters

(1)	F	1	ENTER	: Select Function No.1.
(2)	0	ENTER		: Waiting time → 0 minute
(3)	10	ENTER		: Pump 1 running time → 10 seconds
(4)	8	ENTER		: Pump 2 running time → 8 seconds
(5)	10	ENTER		: Washing time → 10 seconds
(6)	CHG Seq 1-3 ?			: 1 → Without washing
	1	ENTER		: 2 → With washing
				: 3 → Make a working curve.
(7)	Function (0-9) ?		RESET	: "READY" is indicated on the display.

9-4 Measurements of Actual Samples

(1)		Set on a turn-table from the position 1 flasks of samples, S Toluene and R n-Heptane.
(2)	SIZE	
(3)	1	: Input a turn-table's start number.
(4)	Total No.	: Total number of flasks (sample numbers + 2)
(5)	ID	: Sample No.
(6)	SW(g)	: Sample weight
(7)	:	: Input IDO and SWO for S and R respectively.
(8)	START	: Measurement starts.
(9)	START	: Press again if you have set more flask numbers than the turn-table's capacity.

(10)	Samp	: n-Heptane remaining inside the analyzer is drained.
(11)	Samp	: Drain of n-Heptane stops.

APD-500A User's Guide

1st edition: September 2, 2009

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