Spectrum 22ED Spectrophotometer
#FOT 10705

Introduction:

Thank you for your purchase of the Spectrum 22ED spectrophotometer. The Spectrum 22ED is a single beam spectrophotometer and is designed to meet the needs of both students and instructors. Its large and clear LCD digital display, easy operation, and wide wavelength range make the instrument ideal for spectrophotometric experiments in the visible wavelength region of the electromagnetic spectrum.

This user's manual provides information for Spectrum 22ED spectrophotometer, a list of main specifications, and possible labs.

After carefully unpacking the contents, please check the materials against the packing list to ensure that you have received everything in good condition. If you find some part is missing, damaged, or in any way defective, please contact your dealer or sales representative immediately.

However, to keep pace with technological advances, the specifications and operating instructions may be modified or changed as needed. We reserve the right to make design modifications and changes.

Specifications:

	Model 22ED
Spectral slit width	10nm
Wavelength range	360-1000nm
Wavelength accuracy	±3 nm
Wavelength readability	±1 nm
Stray radiant energy	Less than 1%T @340nm
Transmittance	0%T to 100% T
Absorbency	0A to 1.99A
Photometric accuracy	±2.0% T
Power requirement	115/230 VAC; 50-60 Hz automatic
Size	316 (12.4") W x 285 (11.2") D x 195 (7.7") H
Weight	4.5 kg

Unpacking instructions:

Carefully unpack the contents and check the materials against the following packing list to ensure that you have received everything in good condition.

Place the instrument in a suitable location. In order to have the best performance from your instrument, keep it as far as possible from any strong magnetic or electric fields, or any electrical device that may generate high frequency fields. Set the unit up in an area that is free of dust, corrosive gases, and strong vibrations.

Remove any obstructions or materials that could hinder the flow of air under and around the spectrophotometer.

Plug the cord into a grounded outlet. Turn on the spectrophotometer and allow it to warm up for 20 minutes before taking any readings.

Packing List:

1	Spectrophotometer
1	Dust cover
2	Glass cuvettes
1	Black cuvette (occluder)
1	cuvette holder (all cuvettes must be placed in this holder before insertion into sample compartment
1	User's instructions
1	Allen wrench

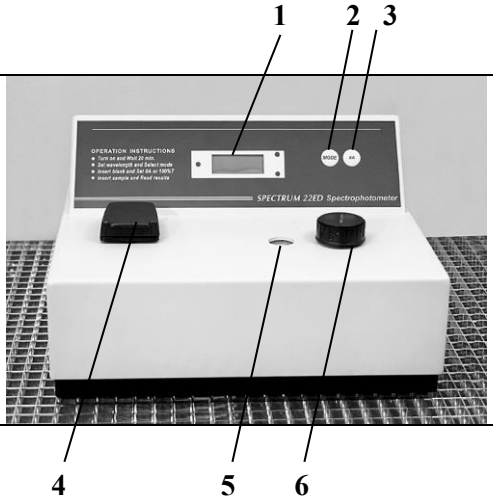
Turning on the unit:

When you turn on the unit, the display will read **1.– – –**

If the power indicator lamp indicates that there is power, but the liquid crystal display remains blank, turn the switch off, unplug the unit and wait 60 seconds. At this point, the unit will have reset. Then, plug the unit back in and turn the unit on.

Operating Features:

1. **LCD Digital Readout**
2. **Mode Selector Key:** Selects Transmittance or Absorbance
3. **OA Control Key:** Used to set the display to 0 A or 100% T
4. **Sample Compartment**
5. **Wavelength Readout**
6. **Wavelength Control Knob:** Used to set the wavelength to the desired value.



Operating Instructions:

1. Plug the instrument into a grounded outlet.
2. Turn the instrument on. Allow it to warm up for at least 20 minutes.
3. Select the wavelength by turning the wavelength control knob.
4. Press **MODE** button to select either Transmittance or Absorbance.
5. Choose matched cuvettes of appropriate path-length for the analytical method you are using. You must use the same path-length cuvettes for all blanks, standards and samples.
6. Fill one of the matched cuvettes with a blank solution (distilled water), wipe the sides of the cuvette with a lint-free tissue, and place the cuvette into the black plastic cuvette holder. Keep in mind that the cuvette holder will be placed in the sample compartment so that the light can pass from right to left across the sample so insert the cuvette appropriately.
7. a. When measuring **Absorbance**, open the sample compartment, place the blank cuvette (distilled water) in the cuvette holder into the well of the compartment so that the green dots are next to each other. Close the sample compartment cover, set the blank by pressing the **0A** key until the display reads .00 A.
   b. When measuring **Transmittance**, open the sample compartment, place blank cuvette (distilled water) in cuvette holder into the well of the compartment so that the green dots are next to each other. Close the sample compartment cover, set the blank by pressing the **OA** key until the display reads 100% T.
9. Remove the cuvette holder from the sample compartment. Remove the cuvette from the cuvette holder.
10. Place the cuvette with the solution to be measured into the cuvette holder. Place the cuvette holder into the sample compartment. Record the reading.
11. Repeat steps 9 – 10 for any additional samples.

Note: With occluder (black cuvette) placed in cuvette holder and inserted into sample compartment, transmittance reading will be 00 and absorbance will display 1.– – – .

Maintenance:

To Replace the Halogen Bulb:

Unit uses a 6V, 10W Halogen bulb (Cynmar #021-00392)
To replace bulb:

1. Shut off unit and unplug it.
2. Remove black screws from lower sides of unit.
3. Slide a small screwdriver into the cap on the wavelength control knob and pop the cap off. Remove brass nut from top of the knob. Slip knob off.
3. With the allen wrench that is provided, loosen set screw on sample compartment. This set screw is at back of compartment approximately one-half inch below cover. Remove sample compartment by pulling it straight upwards.
4. Lift entire top casing off of unit, being careful not to rip out the cables.
5. Set top casing aside.
6. Locate flat black horizontal plate at back left side of unit. Loose two screws at forward position on the plate and slide plate off.
7. Locate halogen bulb.

Note: Do not handle the halogen lamp with bare fingers. Use a piece of tissue paper or cloth when handling the lamp. The oil from your fingers can cause the lamp to burn out prematurely.

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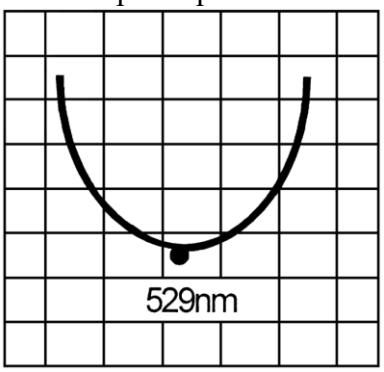
8. Replace the old bulb with a new one. The bulbs have 2-pin connections so they simply pull out and plug in.
9. Slide black plate back into position and tighten the screws that hold it.
10. Place unit casing back into position and reattach the two screws on each side.
11. Reset the sample compartment and tighten the allen head screw.
12. Position knob back onto the post aligning the slot in the fitting to the plastic tab on the knob. Attach the brass nut, tightening it snugly. Replace cap on knob.

Wavelength Calibration:
As usual, the spectrum SP2000-UV spectrophotometer retains its wavelength calibration indefinitely. However, if the instrument receives a severe shock or abuse, use the following methods to check wavelength calibration.

Use of a Didymium filter
The didymium filter has two special absorbance peaks at 529nm and at 808nm. When the instrument is calibrated properly, you will find minimum Transmittance (maximum absorbance) at the range of 529nm (or 808nm) ±2nm.
Note that the specific transmittance values are not important, you are only looking for the wavelength where minimum transmittance (maximum absorbance) occurs.

1. Turn on your unit and allow it to warm up for 20 minutes.
2. Select the % Transmittance operating mode by pressing the **MODE** key.
3. Set the wavelength to 519nm.
4. Insert the cuvette filled with distilled water in position one of the cuvette holder.
5. Press the **100%T** key until the display reads 100.0% T, then remove the cuvette from the unit.
6. Insert the didymium filter in the well of the cuvette holder; record the %T reading on the digital display.
7. Repeat steps 3 through 6 to measure the wavelength at 2nm intervals between 519 and 539nm.
8. The minimum % Transmittance should be obtained between 526nm and 532nm. The wavelength accuracy of the SP2000-UV spectrophotometers is ±2nm.

Wavelength Calibration Curve



Troubleshooting:

Problem		Remedy
Nothing at all appears on the display.		Check that the power indicator lamp is lit. 1. If it is not, check that the cord is connected properly. 2. If it is lit and you still have no display, turn off the unit. Unplug it. Wait 10 seconds, and plug it back in and turn on the switch again. This will allow the unit to reset itself. If you still get no display, call our service department (1-800-223-3517)
Readings differ greatly from expected results.		1. Check wavelength calibration. 2. Check your application, procedure, and sampling technique.
Readings drift.		1. Check your application, procedure, and sampling technique. 2. Check that you are using the correct lamp appropriate to the wavelength you are using. 3. Fumes from sample are affecting the optics of the instrument. 4. Power in the line is fluctuating. 5. Bulb is defective. 6. Instrument is not grounded properly.
Other problems		Call our service department (1-800-223-3517)

Experiments

Experiment 1 — Absorption Spectrum

Materials needed:
Spectrophotometer
0.0500 M Cr^{+3} solution
graph paper

Theory
In this experiment, the absorption spectrum of chromium (III) ion solution will be examined in the range of 350nm to 650nm. You will note two absorption peaks in the visible region, corresponding to the energy level splitting of the 3d electrons in the aqueous complex of the ion. After plotting the spectrum, you can calculate the molar absorptivity from Beer's law at two different wavelengths.

Procedure:
1. Plug the instrument into a grounded outlet.
2. Turn the instrument on and allow the instrument to warm up for at least 20 min.
3. Choose matched cuvettes of appropriate path length for analytical method you are using. You have to use the same path length cuvette for all blanks, standards, and samples.
4. Rinse and then fill a cuvette with distilled water.
5. Rinse a second cuvette with distilled water, followed by rinsing the cuvette twice with your Cr^{+3} solution. Pour rinsings into a waste beaker for later disposal. Fill the cuvette with the Cr^{+3} solution.
6. Wipe the sides of the cuvette filled with distilled water with a lint-free tissue, and place the cuvette in the cuvette holder. Insert cuvette holder into sample compartment.
7. Select the desired operating mode as **ABSORBANCE** by pressing the **MODE** key.
8. Set the unit to a wavelength of 350nm.
9. Close the sample compartment cover. With the blank solution in the light path, calibrate the instrument by pressing the OA key. The LCD will initially display BLA but after a few seconds will read .00.
10. To take an absorbance reading on your sample, remove the cuvette holder. Remove blank cuvette from holder. Place cuvette containing sample into the cuvette holder. Insert cuvette holder into sample compartment and record the absorbance of your sample.
11. Repeat steps 8 to 10 for wavelengths from 360nm to 650nm in 10nm intervals. Make sure the unit is calibrated with the 0A key at each new wavelength.

Calculations:

1. On a sheet of graph paper, label the x-axis wavelength, and mark it from 350 to 650nm in 10nm intervals. Label the y-axis absorbance and mark it in equal intervals from 0 to a convenient whole number above your highest data point.
2. Plot the absorbance for each wavelength measured.
3. Connect the data points with a smooth curve.
4. Determine the wavelengths at which the chromium (III) solution absorbs the most and the least. From the graph, determine the absorbance at each of these wavelengths.

Absorption Spectrum Report

Wavelength	Absorbance	Wavelength	Absorbance
350	_____	**510**	_____
360	_____	**520**	_____
370	_____	**530**	_____
380	_____	**540**	_____
390	_____	**550**	_____
400	_____	**560**	_____
410	_____	**570**	_____
420	_____	**580**	_____
430	_____	**590**	_____
440	_____	**600**	_____
450	_____	**610**	_____
460	_____	**620**	_____
470	_____	**630**	_____
480	_____	**640**	_____
490	_____	**650**	_____
500	_____		

Calculations:

Absorption maximum wavelength: _____ **nm** **Absorbance:** _____

Absorption minimum wavelength: _____ **nm** **Absorbance:** _____

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Experiment 2 **Beer's Law**

Materials Needed:

0.100 M Cr^{+3} solution
unknown solution
spectrophotometer
graph paper
4 Erlenmeyer flasks
100 mL volumetric flask
50 mL buret

Theory:

We can study the quantitative relationships between the amount of light absorbed by chromium (III) ion and its concentration. The relationship between absorbance and concentration is given by Beer's law A = a b c, where A is absorbance, a is absorptivity, b is path width, and c is concentration. We will need to measure the absorbance of several samples of varying but known concentrations of chromium (III), as well as one sample of unknown concentration. We can then plot concentration versus absorbance. From the graph, we can then determine the concentration of the unknown solution.

Procedure:

1. Rinse the buret twice with small volumes of the stock 0.100 M Cr^{+3} solution. Collect the rinsings in a waste container for disposal later. Fill the buret with the chromium (III) solution.
2. Add 5.0 mL of the stock solution from the buret into a 100 ml volumetric flask. Add distilled water to the mark and stopper the flask. Mix this diluted solution thoroughly.
3. Season one of the Erlenmeyer flasks with small portion of this diluted standard, discarding the rinsings into the waste container. Pour the diluted standard from the volumetric flask into this Erlenmeyer flask and stopper the flask. Calculate the concentration of this first standard and label it appropriately. Record the result on the report sheet.
4. Rinse the volumetric flask with distilled water and repeat step 3 using 10.0mL of the stock from the buret. Repeat step three twice more, once using 15.0mL, and once using 20.0mL.
5. After the four standard solutions have been prepared, rinse the volumetric flask with water.
6. Obtain the unknown solution from your instructor.
7. Refer to an absorption spectrum of chromium (III) and choose a wavelength at which the ion absorbs most strongly. [You can refer to the results from Experiment 1 or can refer to a reference work to determine this wavelength.] Set your spectrophotometer to the chosen wavelength. Fill a clean cuvette with distilled water, wipe the cuvette clean, and place it in position 1 of the cuvette holder. Set the **MODE** to absorbance.
8. Close the sample compartment cover. With the blank solution in the light path, calibrate the instrument by pressing the 0A key. The LCD will initially display BLA but after a few seconds will read .00A.
9. Fill a second cuvette with the first standard (or empty the cuvette of distilled water, rinse twice with the first standard to keep from diluting your standard). Insert the cell into a sample compartment. Align the cell with the light beam and read the absorbance of this first standard. Record the result.
10. Repeat the process with the other three standards and with the unknown solution.

Report Sheet

Data:

Solution	Concentration	Absorbance
Standard 1		
Standard 2		
Standard 3		
Standard 4		
Unknown solution		

Calculations:

1. Graph concentration versus absorbance, graphing concentration on the x-axis and absorbance on the y-axis.
2. Plot absorbance versus concentration for the four standard solutions. Draw the best straight line through the four points and the origin of the graph. This is the standard curve for Beer's law.
3. From the measured absorbance of the unknown sample, read the graph to determine the concentration of chromium (III) ion in the unknown. Record this on your report sheet.

Experiment 3 **Reaction Kinetics**

Materials Needed:

0.010 M Cr^{+3} solution
0.20 M Na_2EDTA (EDTA disodium) (pH 6.15)
0.010 M Na_2CO_3 (aq)
Graduated cylinder
5 Erlenmeyer flasks
graph paper
2 50mL burets
hotplate

Theory

Chemical reactions occur as a result of collisions between atoms, molecules, or ions of the reactants. Kinetics deals with the rate of chemical reactions, with the factors that influence those rates, and the mechanism of the reaction. The rate of a reaction can be determined experimentally by measuring the change in concentration of the reacting species or the products as a function of time.

In this experiment, you will measure the effect of one reactant (Cr^{+3}) on the formation of a colored complex ion formed between chromium (III) and ethylenediamine-tetraacetic acid (EDTA). You will measure the appearance of the complex with the spectrophotometer. You will also observe the effect of temperature on the rate of the reaction, and the effect of a catalyst (carbonate ion) on the rate.

The rate of a reaction is defined as the rate of disappearance of a reactant or appearance of a product. The rate is related to the concentration of the reactants through the rate law. For example, if we consider the following reaction:

$$a\,A + b\,B \rightarrow c\,C$$

Rate = $k\,[A]^x\,[B]^y$ where k is the rate constant, [A] and [B] are the molar concentrations of the reactants, and x and y are the order of the reaction with respect to A and B.

Procedure:

1. Rinse five Erlenmeyer flask, six cuvettes, two burets and the graduated cylinder with distilled water.
2. Rinse one of the burets with a small amount of the stock Cr^{+3} solution to remove water. Allow the rinse to drain through the stopcock into a waste container. Fill the buret with the stock chromium (III) solution.
3. Rinse and fill the second buret with distilled water.
4. From the burets, add the following volumes of each solution to five clean, dry Erlenmeyer flasks:

FLASK NUMBER	mL Cr^{+3} (aq)	mL H_2O
1	5.0	20.0
2	10.0	15.0
3	15.0	10.0
4	20.0	5.0
5	10.0	10.0

Calculate the concentrations of chromium (III) in flasks 1 through 4 and record the results on the report sheet.
5. To flask 5, add 5 mL of 0l010 M Na_2CO_3 (aq) from a graduated cylinder.
6. Set the wavelength of the spectrophotometer to 545 nm. Adjust the **MODE** to read **Absorbance**. Fill a cuvette with distilled water and insert it into the cuvette holder. Calibrate the meter by pressing the 0A key. After a few seconds it should read .00. Remove the distilled water cuvette and set it aside.

Note: The following steps require you to time all additions and measurements at two minute intervals. You need to read through the entire procedure and understand the entire procedure before starting.

7. At time = 0.0 minutes, use a graduated cylinder to add 25 mL of the EDTA solution to flask 1. Swirl the contents, rinse a cuvette with the solution to season it, then fill the cuvette. Label the cell and set it aside. Place this flask on a hotplate with the setting at LOW. (Do not allow the solution to boil, as it will start to decompose.)
8. At time = 2.0 min, 4.0 min, 6.0 min, and 8.0 min repeat step 7 with flasks 2, 3, 4, and 5 and cuvettes 2, 3, 4, and 5. As you did with flask 1, place flasks 2, 3, and 4 on the hotplate but do **not** heat the solution in flask 5.
9. At time = 10.0 minutes, insert cuvette 1 into the sample holder and measure the absorbance. Record the results on the report sheet. Remove the cell and set it aside.
10. At time = 12.0 min, 14.0 min, 16.0 min, and 18.0 min, measure the absorbance of the remaining four solutions, in numerical order, at two minute intervals. Record the results on the report sheet. After removing cuvette 5 from the sample compartment, insert the cell containing distilled water. Adjust the meter by pressing the 100%T/0A key.
11. At time = 20.0 min until time = 110.0 min, repeat the measurements at two minute intervals, so that each solution is measured every ten minutes. Record the results on the report sheet. Adjust the 100%T/0A reading with distilled water cuvette after every cycle of the five solutions.
12. At the end of the measurements, remove the flasks from the flasks from the hotplate, and allow them to cool to room temperature. Measure the absorbance of each of these solutions when they are cool.

Calculations:
1. On a sheet of graph paper, label the x-axis as concentration and the y-axis as absorbance. Mark off intervals appropriate to the data you have collected.
2. Plot the absorbance vs. concentration for the heated solutions in flasks 1 through 4. Draw the best fit straight line through the four points and the origin.
3. On a second sheet of graph paper, label the x-axis time and the y-axis absorbance. Mark off intervals appropriate for all five of the timed runs.
4. For each of the five sample runs, plot, the absorbance value versus time. Time is measured from the time the EDTA is added to the solution. (0 to 100 minutes in 10 minute intervals) Draw a smooth curve through the data set for each of the five solutions.
5. Using the second graph, determine the order of the reaction with respect to Cr^{+3}.

Report Sheet

Data

Time (in min)	Absorbance Cuvette Number				
	1	2	3	4	5
10.0					
20.0					
30.0					
40.0					
50.0					
60.0					
70.0					
80.0					
90.0					
100.0					

Flask	Concentration	Absorbance
1		
2		
3		
4		

Results:

1. Determine the order of the reaction with respect to Cr^{+3}.
2. Discuss the effect of the catalyst (flask 5).
3. Discuss the effect of temperature.
4. Discuss the effect of concentration.

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Instructors Notes for the Experiments

Experiment 1.

Chromium (III) solution:

To make the 1 liter of the 0.0500 M stock solution, add 20.0 grams of $Cr(NO_3)_3 \bullet 9 H_2O$ then fill to 1 liter mark. Each student group will need approximately 20 mL of the stock solution.

Experiment 2.

Chromium (III) solution:

To make 1 liter of the 0.100 M stock solution, dissolve 40.0 grams of $Cr(NO_3)_3 \bullet 9 H_2O$, then fill to 1 liter mark. Each student group will need approximately 60 mL of the stock solution.

The Beer's law plot should be linear over the concentration range used in this experiment.

The 0.100 M Cr^{+3} stock solution can be used for the unknown solution by varying the volume given to each student group. The volumes should range between 5 mL and 10 mL so that the final concentrations, after dilution, are in the range of 0.0050 to 0.0200M.

Experiment 3.

Chromium (III) solution:

To make 1 liter of the 0.0100 M stock solution, dissolve 4.0 grams of $Cr(NO_3)_3 \bullet 9 H_2O$, then fill to 1 liter mark. Each student group will need approximately 70 mL of the chromium (III) solution.

EDTA solution

To make 1 liter of the 0.200 M EDTA solution, dissolve 74.45 grams of EDTA disodium salt, then fill to 1 liter mark. Each student group will need approximately 135 mL of the EDTA solution.

Sodium carbonate solution

To make 1 liter of the 0.010 M Na_2CO_3 solution, dissolve 1.06 grams, then fill to the 1 liter mark. Each student group will need approximately 5 mL of the sodium carbonate solution.

The reaction is first order (x = 1) in chromium (III).

Limited Warranty

Purchase of items branded Cynmar® are warranted against defects in workmanship and materials for 90 days from the original purchase date. Should there be a defect or malfunction of the product, Cynmar® will repair or replace the product (at its option) free of charge excluding shipping charges, which remain the responsibility of the Purchaser. This limited warranty is void if the product has been subjected to damage, unreasonable use, improper service, modification, or other causes not arising from defects in original materials or workmanship.

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