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AUTHOR Bonta, John L.; Davidson, Arnold C.
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ABSTRACT

This document is an instructional module package prepared in objective form for use by an instructor familiar with the determination of color in water using the visual comparison method and the spectrophotometric method. Included are objectives, instructor guide, student handouts, and transparency masters. A videotape is also available from the author. The module addresses the importance of color in water, preparation of standards, relationship of hue to wavelength, use of a spectrophotometer, measuring transmittance, and calculating and reporting results. (Author/RH)

DETERMINATION OF COLOR IN WATER

Training Module 5.250.2.77

Prepared for the

Iowa Department of Environmental Quality
Wallace State Office Building
Des Moines, Iowa 50319

by

John L. Bonte
Developer
Arnold C. Davidson
Project Director
Clinton Community College
1000 Lincoln Boulevard
Clinton, Iowa 52732

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September, 1977

Module No:

Module Title:
The Determination of Color in Water

Submodule Title:

Approx. Time:
5 hours

Topic:
Summary

Instructional Objective:

Upon completion of this module the participant should be able to:

1. Determine the pH of a water sample.
2. Determine the color in "Color Units" in a water sample by the visual comparison method.
3. Adjust the pH of a water sample to 7.6 with H_2SO_4 or NaOH.
4. Determine the color characteristics of a water sample using the spectrophotometric method.

Instructional Aids:
Transparencies Col - Co9
Color/threshold odor video tape.

Instructional Approach:

Practice, lecture/discussion, video tape viewing

References:

1. "Standard Methods for the Examination of Water and Waste Water."
2. Operator's Manual for the Bausch and Lomb spectronic 20.

Class Assignments:

Module No:

Module Title:
The Determination of Color in Water

Submodule Title:
Visual Comparison Method

Approx. Time:
5 hours

Topic:
Importance of Color measurement

Instructional Objective:

Upon completion of this module the participant should be able to:

1. Explain the origin of color in natural water.
2. Explain the difference between true color and apparent color.
3. Briefly explain the difference between the visual comparison and the spectrophotometric method for the determination of color.

Instructional Aids:

Transparency Co-1, Color/odor video tape

Instructional Approach:

Lecture/discussion
T.V. viewing

References:

Standard Methods, pp. 64-68.

Class Assignments:

None

Module No: Co	Topic: Importance of color measurement

Instructor Notes:	Instructor Outline:
Color/odor video tape	1. Show tape (1st half), answer questions
Transparency Co-1 Sources of color in water	2. Color sources a. metal ions b. organic material c. industrial waste
	3. True and apparent color: a. apparent: true plus from turbidity b. true: color of filtered sample
	4. Comparison method measures intensity of color only Spectrophotometric measures intensity and hue of color.

Module No:	Module Title: The Determination of Color in Water
	Submodule Title: Visual Comparison Method
Approx. Time: .5 hours	Topic: Preparation of standards

Instructional Objective:

Upon completion of this module the participant should be able to:

1. Prepare a stock standard color solution containing 500mg Pt and 250 mg Co per liter.
2. Dilute the stock solution properly to produce a set of standards

Instructional Aids:

Transparency Co2/ Dilution of color standard.

Instructional Approach:

Laboratory practice

References:

Standard Methods, p. 64-66.

Class Assignments:

None

Module No: Co	Topic: Preparation of Standards - Visual Comparison
Instructor Notes:	Instructor Outline:
1. STock solution 1.246 K_2PtCl_6 1.00g $CoCl_2 \cdot 6H_2O$ 100 m l HCl dilute to 1 liter	1. Prepare stock solution - 500 mg/l Pt, 250 mg/l Co. This is a solution which contains 500 color units should be done prior to session or in groups.
2. Transparency Co-2 Dilution of color standards	2. From stock solution prepare standards according to dilution chart directly in nessler tubes.

Module No:	Module Title: The Determination of Color in Water.
	Submodule Title: Visual Comparison Method
Approx. Time: .5 hours	Topic: Measurement of pH

Instructional Objective:

Upon completion of this module the participant should be able to:

1. Operate a simple pH meter.
2. Standardize a pH meter given a buffer solution.
3. Determine the pH of a water sample.
4. Explain precautions which should be taken in the care of a meter and its electrodes.

Instructional Aids:

None

Instructional Approach:

Laboratory practice

References:

Standard Methods, p. 464

Class Assignments:

None

Module No: Co	Topic: Measurement of pH

Instructor Notes:	Instructor Outline:

1. Demonstrate operation of pH meter

2. Standardize with pH7 buffer

3. Have students determine the pH of the colored water sample and record

4. Electrodes should be washed with distille water and blotted dry between measurements. Electrodes should be stored in distilled water.

Module No:

Module Title:
The Determination of Color in Water

Submodule Title:
Visual Comparison Method

Approx. Time:
.5 hours

Topic:
Measurement and Calculation

Instructional Objective:

Upon completion of this module the participant should be able to:

1. Compare the color intensity of a sample with that of the standards.
2. Determine the number of color units a sample contains.
3. If there are more than 70 color units properly dilute the sample and determine color units.
4. Properly report color.

Instructional Aids:

Transparency Co3: Reporting visual color.

Instructional Approach:

Laboratory practice

References:

Standard Methods, p. 66

Class Assignments:

None

Module No:	Topic:
Co	Measurement and Calculation visual comparison

Instructor Notes:	Instructor Outline:
Transparency Co-3 Reporting visual color	1. Properly observe sample and determine standard of closest color. 2. Determine color units to closest 1 below 50, 5 above 50. 3. If above 70 dilute 1:1 or more and report color 4. Calculate color of diluted sample: $\text{color units} = \frac{(\text{color units measured}) \times 50}{\text{ml sample diluted}}$ 5. Report color units and pH of sample

Module No:

Module Title:
The Determination of Color in Water

Submodule Title:
Spectrophotometric Method

Approx. Time:
.25 hours

Topic:
Relation of Hue to Wavelength

Instructional Objective:

Upon completion of this module the participant should be able to:

1. Given a range of wavelengths of light determine the hue of color of light given a wavelength/hue chart.

Instructional Aids:

Transparency Co4: wavelength/hue chart

Instructional Approach:

Lecture/discussion

References:

Standard Methods, p. 68

Class Assignments:

None

Module No:	Topic:
Co	Relation of hue to wavelength-Sepctro method

Instructor Notes:	Instructor Outline:
Transparency Co-4 Wavelength hue chart	1. Point out relation between hue and color from chart a. the hue refers to color of light transmitted

Module No:	Module Title: The Determination of Color in Water.
	Submodule Title: Spectrophotometric Method
Approx. Time: 0.25 hours	Topic: Safety

Instructional Objective:

Upon completion of this module the participant should be able to:

1. Locate the following in the laboratory and demonstrate proper use: emergency shower, fire extinguisher, eye wash, first aid kit.
2. Select and use safety glasses, lab coat or apron and gloves in the appropriate situation.
3. Recognize the hazards of sulfuric acid and sodium hydroxide solutions.

Instructional Aids:

Handout of safety rules for the laboratory.

Instructional Approach:

Lecture/discussion

References:

Basic laboratory skills module.

Class Assignments:

Read lab safety rules

Module No: Co	Topic: Safety - Spectrophotometric
Instructor Notes:	Instructor Outline:

1. Point out the location of safety appliances in laboratory

2. Safety glasses should be worn when adjusting the pH

3. The only dangers are those of shock or burns when handling NaOH or H_2SO_4 solutions

Module No:

Module Title:
The Determination of Color in Water

Approx. Time:
0.5 hours

Submodule Title:
Spectrophotometric Method

Topic:
Adjustment of pH

Instructional Objective:

Upon completion of this module the participant should be able to:

1. Select either solutions of NaOH or H_2SO_4 for pH adjustment.
2. Using a pH meter and NaOH or H_2SO_4 solution adjust the pH of a water sample to 7.6.

Instructional Aids:

Transparency Co6: Relation of acidity/Basicity to pH

Instructional Approach:

Laboratory practice

References:

Standard Methods, p.66

Class Assignments:

None

Module No: Co	Topic: Adjustment of pH

Instructor Notes:	Instructor Outline:
Transparency Co-6 Relation of acidity/ basicity to pH	1. If the pH is above 7.6 use H_2SO_4. If it is below 7.6 use NaOH for pH adjustment. 2. Add acid or base dropwise with stirring. If the water is not buffered naturally it will be easy to over-shoot. If so the pH can be re-adjusted with base if acid was used originally or with acid if base was used originally.

Module No:

Module Title:
The Determination of Color in Water

Submodule Title:
Spectrophotometric Method

Approx. Time:
0.5 hours

Topic:
Filtration

Instructional Objective:

Upon completion of this module the participant should be able to:

1. Set up a filtration system for color determination.
2. Filter a sample of turbid water to produce a clear sample.

Instructional Aids:

Transparency Co5: Filter diagram

Instructional Approach:

Laboratory practice

References:

Standard Methods, pp. 66, 67.

Class Assignments:

None

Module No: Co	Topic: Filtration - Spectrophotometric
Instructor Notes:	Instructor Outline:
Transparency Co-5 Filter Diagram	1. Students will set up filter system and pre-coat. 2. As a second step they should filter a sample of colored turbid water.

Module No:

Module Title:
The Determination of Color in Water

Submodule Title:
Spectrophotometric Method

Approx. Time:
0.25 hours

Topic:
Use of the Spectrophotometer

Instructional Objective:

Upon completion of this module the participant should be able to:

1. Operate a spectrophotometer such as the Bausch and Lomb Spectronic-20:
   a. turn on and warm-up
   b. set wavelength
   c. set zero
   d. set 100% T
2. Briefly explain how a spectrophotometer works.

Instructional Aids:

Transparency Co6: condensed spectrophotometer operating instructions.
Transparency Co7: diagram of spectrophotometer

Instructional Approach:

Lecture/demonstration

References:

Operator's Manual for spectrophotometer used

Class Assignments:

Module No: Co	Topic: Use of Spectrophotometer

Instructor Notes:	Instructor Outline:
Transparency Co-7 Diagram of spectrophotometer Transparency Co-10 Condensed spectrophotometer, operating instructions	1. Demonstrate setting of wave length, setting of zero, setting of 100% T 2. From transparency demonstrate the basic parts: light source, lenses and mirrors, phototube and sample compartment

Module No:

Module Title:
The Determination of Color in Water

Approx. Time:
0.5 hours

Submodule Title:
Spectrophotometric Method

Topic:
Measurement of % T

Instructional Objective:

Upon completion of this module the participant should be able to:

1. measure the percent transmittance of a water sample at the 10 sets of ordinate wavelengths.

Instructional Aids:

Transparency Co8: Selected ordinates for color.

Instructional Approach:

Laboratory practice

References:

Standard Methods p. 67,

Class Assignments:

None

Module No: Co	Topic: Measurement of % T - Spectrophotometric

Instructor Notes:	Instructor Outline:
Transparency Co-8 Selected ordinates for color	1. Make available a colored sample a. set wavelength, zero, 100% T for each reading b. determine % T for each of the 10x3=30 ordinates c. record values of %T

Module No:	Module Title: The Determination of Color in Water.
	Submodule Title: Spectrophotometric Method
Approx. Time: 0.75 hours	Topic: Calculation and Reporting of Results

Instructional Objective:

Upon completion of this module the participant should be able to:

1. Determine tristimulus values X, Y, and Z from wavelength/ % T data.
2. Determine trichromatic coefficients x and y from X, Y, and Z values.
3. Determine dominate wavelength and purity from x, y and the chromaticity diagram.
4. Report all data used in expressing spectrophotometric color results.

Instructional Aids:

Transparency Co9: Chromaticity diagrams

Instructional Approach:

Lecture/discussion

References:

Standard Methods, p.68, 69.

Class Assignments:

None

Module No: Co	Topic: Calculation and Reporting results

Instructor Notes:	Instructor Outline:
Transparency Co-9 Chromaticity diagram	1. Sum values in columns X, Y, Z, multiply by "Factors" 2. $x=\frac{X}{X+Y+Z}$ $y=\frac{Y}{X+Y+Z}$ 3. Values of purity, dominant wavelength are determined from x, y and chromaticity diagram. 4. Report for sample and sample with adjusted pH: a. dominant wave lengths(from 3) b. hue(from color chart) c. luminance(Y) d. purity(from 3) e. instrument(spectronic 20) f. number of ordinates(10) g. band width(from mfg literature)

Exam Questions

The Determination of Color in Water
Visual Comparison Method
Importance of Color Measurement

1. Which of the following is not a source of color in water?
   a. industrial wastes
   b. metallic ions
   c. chloride ion
   d. plankton

2. If turbidity is present and has not been removed prior to visual comparison, how should it be reported?

3. The chief difference between the visual comparison method and the spectrophotometric method is that the spectrophotometric method makes use of an instrument called a _____.

Preparation of Standards

4. What two metal ions are used as color comparison standards?

5. Each color standard will have a final volume of _____ ml.

Measurement of pH

6. What does a pH meter measure?

7. A buffer solution is used to _____ the pH meter.

8. The pH of a solution is determined by immersing the _____ in the solution.

9. Are the electrodes used in pH measurement sturdy or fragile?

Measurement and Calculation

10. In the comparison of sample color and standard color should the tubes be observed vertically or perpendicular to the tubes?

11. What is the maximum number of color units that should be observed without dilution?

12. If 10 ml of a sample are taken for dilution to 50 ml in a Nessler tube and the diluted sample contains 40 color units, calculate the number of color units in the original sample.

13. In addition to color units, what other piece of data should be reported when reporting color?

Spectrophotometric Method
Relation of Hue to Wavelength

14. True or False: The wavelength of light absorbed by a sample is related to the hue of color of the sample.

Safety

15. When the fire extinguisher is used, the horn should be pointed

at the top of the fire or at the base of the fire?

16. When concentrated acid or base is used to adjust pH, what should be worn to protect the eyes?

17. Is it a good idea to mix concentrated H_2SO_4 with concentrated NaOH?

Adjustment of pH

18. A solution has a pH of 9.1. Would you use H_2SO_4 or NaOH to adjust the pH to 7.6?

19. What instrument is used to measure the success of pH adjustment?

Filtration

20. A filtration apparatus consists of two flasks. One is for collection of the sample. What is the other for?

21. In the filtration procedure, what is the purpose of the precoat?

Use of the Spectrophotometer

22. How long should the spectrophotometer be allowed to warm up prior to making measurements?

23. What is the purpose of the spectrophotometer photo tube?

Measurement of % T

24. For each ordinate wavelength, how many transmittance values should be recorded?

Calculation and Reporting Results

25. The sum of the ordinate values under "X" is 500(for ten ordinates). The "factor" is 0.09806. Calculate the tristimulus value, X.

26. If X=.053, Y=0.51, Z=.048, calculate the value of the trichromic coefficient x.

27. Calculate the value of y in question 26.

28. Is "hue" expressed as a number or as a word?

COLOR

EQUIPMENT AND SUPPLIES LIST

1. spectrophotometer
2. spectrophotometer cells
3. analytical balance
4. potassium hexachloroplatinate (IV) K_2PtCl_6
5. cobalt (II) chloride hexahydrate $CoCl_2 - 6H_2O$
6. concentrated hydrochloric acid
7. 100 ml graduated cylinder
8. 1 ℓ volumetric flask
9. 12 - 50 ml nessler tubes
10. 2, 5, 10 ml graduated pipets
11. distilled water
12. sodium hydroxide (NaOH)
13. polyethylene bottle
14. 1 ℓ beaker
15. pH meter
16. pH 7 buffer
17. 2 - 150 ml beaker
18. concentrated sulfuric acid H_2SO_4
19. centrifuge
20. "filter aid" - celite 505
21. filter crucible, holder, 2 filter flasks, 3 way stopcock, vacuum tubing
22. chromaticity diagrams, or transparency Co-4

The Determination of Color in Water

Laboratory Procedure

I. Preparation of Standards

A. Obtain all equipment, supplies, and chemicals listed in "equipment" handout. Turn spectrophotometer on for warm-up.

B. Stock Standard: Accurately (analytical balance) weigh 1.246 grams potassium hexachloroplatinate (IV) chloride hexahydrate (K_2PtCl_6-$6H_2O$) and 1.00g cobalt (II) chloride ($CoCl_2$-$6H_2O$) and transfer to a 1 l volumetric flask. Add 100 ml concentrated HCl. Mix to dissolve and dilute to the mark with distilled water. Mix well.

C. Label twelve 50 ml Nessler tubes: 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 60, 70 and add to them respectively 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5, 5.0, 6.0, 7.0 ml stock standard solution. Dilute each to the mark with distilled water. Place a rubber stopper on top of each for storage.

D. Concentrated NaOH: Dissolve 625g solid NaOH (sodium hydroxide) in distilled water (800 ml). Allow to stand 48 hours. Filter. Transfer to a polyethylene bottle. Label: 15N NaOH concentrated.

II. Visual comparison method

A. With a standardized pH meter, measure the pH of the water sample to be measured. Record this value on the data sheet.

B. Fill a nessler tube to the 50ml mark with the sample. Look vertically downward through the tube and compare with the standard. A white surface should be used. Determine the two standards whose colors are just darker and lighter than the sample. Record the color units of each. Record to nearest 0.1 the color of the sample.

C. If the sample is too dark add 10ml to the nessler tube. Dilute to the mark with water. Compare as in B above. The color will be 5 times the color of the diluted sample.

D. Comment on the turbidity or other problems.

III. Spectophotometric determination

A. Set up

1. pH adjustment: Choose a well mixed sample. Pour 50 ml into each of two 150 ml beakers. Label one beaker

"adjusted"; the other "raw". Measure the pH. If the pH is less than 7.6 add concentrated NaOH dropwize with mixing to the "adjusted" beaker until the pH is 7.6. If the pH is greater than 7.6, add concentrated H_2SO_4 dropwize until the pH reaches 7.6. Record the original pH.

2. Filtration: Treat each sample as follows: Centrifuge sample. Retain the liquid, discard the sediment. Add 10ml to 0.1g "filteraid". Mix. Pour into filter crucible and direct stream to waste flask. Mix .040g filteraid with 35ml sample. Pour into filter crucuible directing filtrate to waste flask. When filtrate is clear, direct to clean flask. Collect 25 ml filtrate.

B. Spectrophotometric measurement. Set 0% T with sample compartment empty. Set 100% T with absorption cell filled with distilled water. Repeat this for each measurement. Rinse another curette with sample and fill. Determine first wavelength and set. Insert sample in compartment. measure % T. Repeat for each ordinate value X, Y, Z for each of 10 ordinates listed. Repeat and record for other sample.

C. Calculation of results.

1. Sum each of the three columns X, Y, Z.

2. Multiply each sum by the indicator factor to get tristimulus values.

3. Calculate trichromic coefficients X, Y
   $x = X/(X+Y+Z)$
   $y = Y/(X+Y+Z)$

4. Locate the ordered pair x,y on the chromaticity diagram (transparency Co-3 prepared as a hand-out).

5. Determine the hue from the dominant wavelength and the color chart (transparency Co-4 prepared as hand-out).

6. Report instrument make and spectral bandwidth (from mfg literature or setting)

D. Comment on any sources of error, sampling or changes in procedure.

Color Determination

Data Sheet

Sample no._____

I. Visual comparison
pH_____

standard just darker _____units
Standard just lighter _____units
color of sample _____units
was sample diluted?
If so multiply color of diluted sample by 5.0 to obtain color _____units. Was this "apparent" color?

Comment:

II. Spectrophotometric
Original pH_____
Adjusted to pH_____

Ordinate no.	X Raw	X nm	X pH adjusted	Y Raw	Y nm	Y pH adjusted	Z Raw	Z nm	Z pH adjusted
1		433.5			489.5			422.2	
2		461.2			515.2			432.0	
3		544.3			529.8			438.6	
4		504.1			541.4			444.4	
5		577.4			551.8			450.1	
6		588.7			561.9			455.9	
7		599.6			572.5			462.0	
8		610.9			584.8			468.7	
9		624.2			600.8			477.7	
10		645.9			626.3			495.2	
Sum									
Factor	X0.09806		X0.09806	X0.1000		X0.1000	X0.11814		X0.11814
Tristimulus values				% Luminance		% Luminance			

Raw Sample	pH Adjusted Sample
$x = \frac{X}{X+Y+Z} =$ _____	$x =$ _____
$y = \frac{Y}{X+Y+Z}$ _____	$y =$ _____
Purity= _____ %	Purity= _____ %
dominant wavelength _____ nm	dominant wavelength _____ nm
hue _____	hue _____

Instrument (brand):
10 ordinants
bandwidth _____ nm.

Comments:

Analyst _____ Date _____

TRANSPARENCY CO1

Sources of Color in Water

1. Metallic ions
   a. iron
   b. manganese
2. Humus
3. peat materials
4. Plankton
5. Weeds
6. Industrial wastes

TRANSPARENCY CO2

Dilutions of Color Standards

ml standard(500 units) to be diluted to 50 ml	color units of diluted sample
0.5	5
1.0	10
1.5	15
2.0	20
2.5	25
3.0	30
3.5	35
4.0	40
4.5	45
5.0	50
6.0	60
7.0	70

TRANSPARENCY C03

Reporting Visual Color

In reporting color determined by visual comparison, report:

Color units

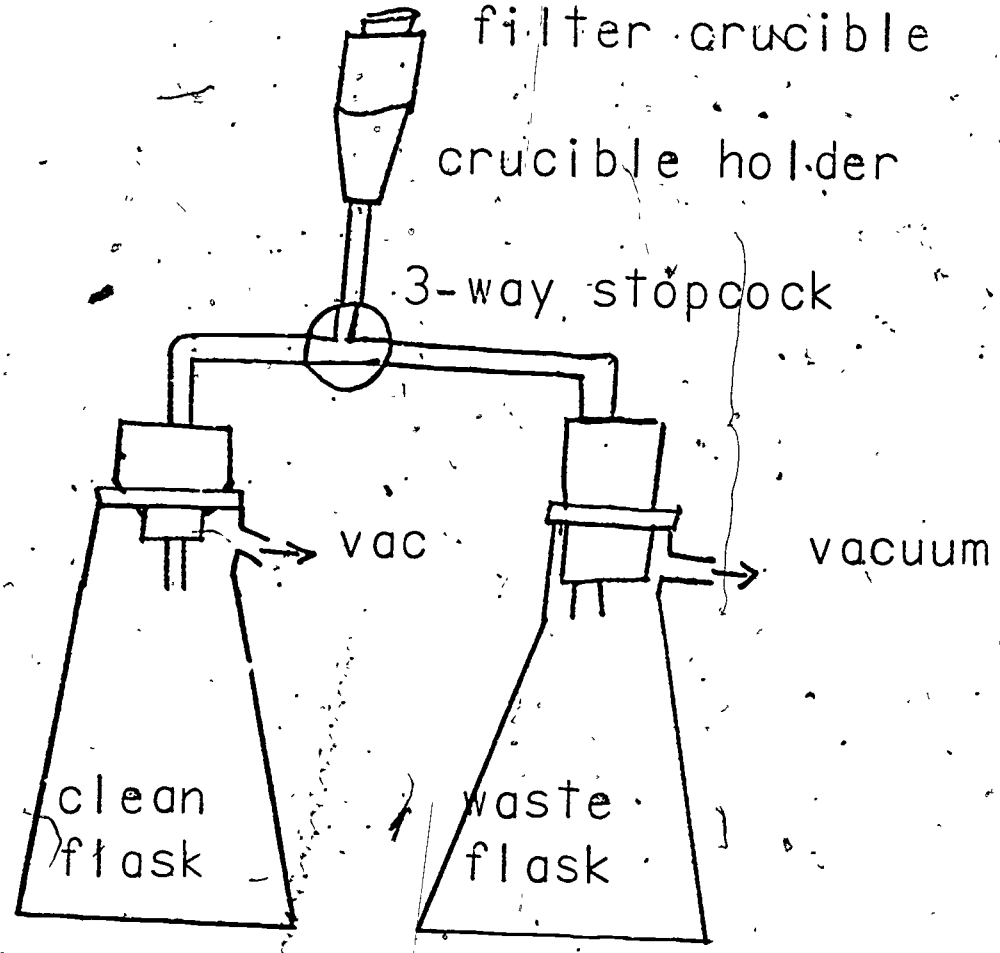
pH of sample

If the sample is furbid report as "apparent color", or filter and report color of filtrate

TRANSPARENCY C04

Wavelength-hue Chart

wavelength range (nm)	hue
400-465	violet
465-482	blue
482-497	blue-green
497-530	green
530-575	greenish-yellow
575-580	yellow
580-587	yellowish-orange
587-598	orange
598-620	orange-red
620-700	red
400-530c	blue-purple
530-700c	red-purple



TRANSPARENCY C05
Filter Diagram

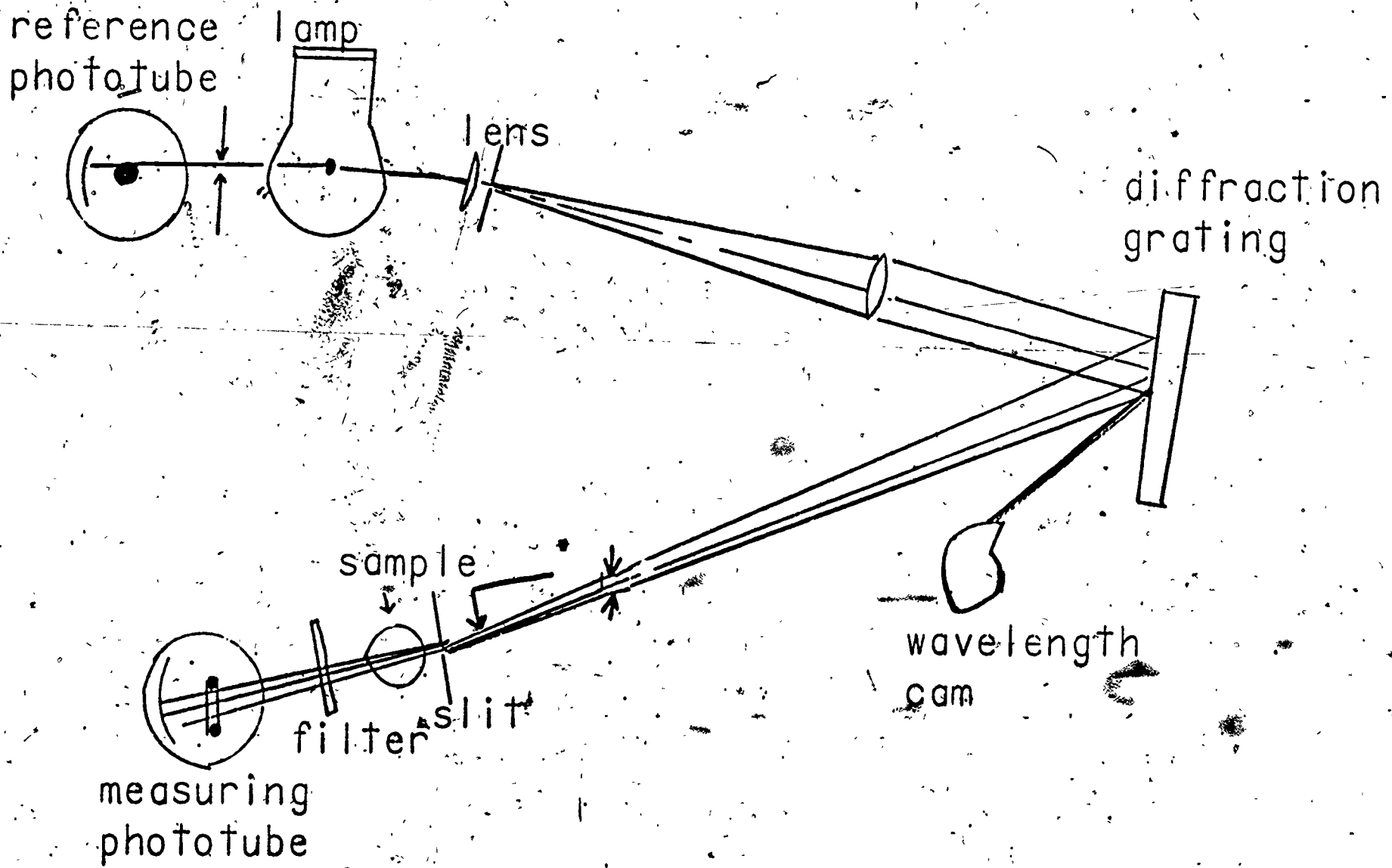
TRANSPARENCY C06

Relation of pH to Hydrogen and Hydroxide Ion Concentration (Molarity)

pH	OH^-	H^+	
1	10^{-13}	10^{-1}	ACIDIC
2	10^{-12}	10^{-2}	
3	10^{-11}	10^{-3}	
4	10^{-10}	10^{-4}	
5	10^{-9}	10^{-5}	
6	10^{-8}	10^{-6}	
7	10^{-7}	10^{-7}	
8	10^{-6}	10^{-8}	
9	10^{-5}	10^{-9}	BASIC
10	10^{-4}	10^{-10}	
11	10^{-3}	10^{-11}	
12	10^{-2}	10^{-12}	
13	10^{-1}	10^{-13}	
14	1	10^{-14}	

TRANSPARENCY C07

Spectrophotometer diagram



TRANSPARENCY C08

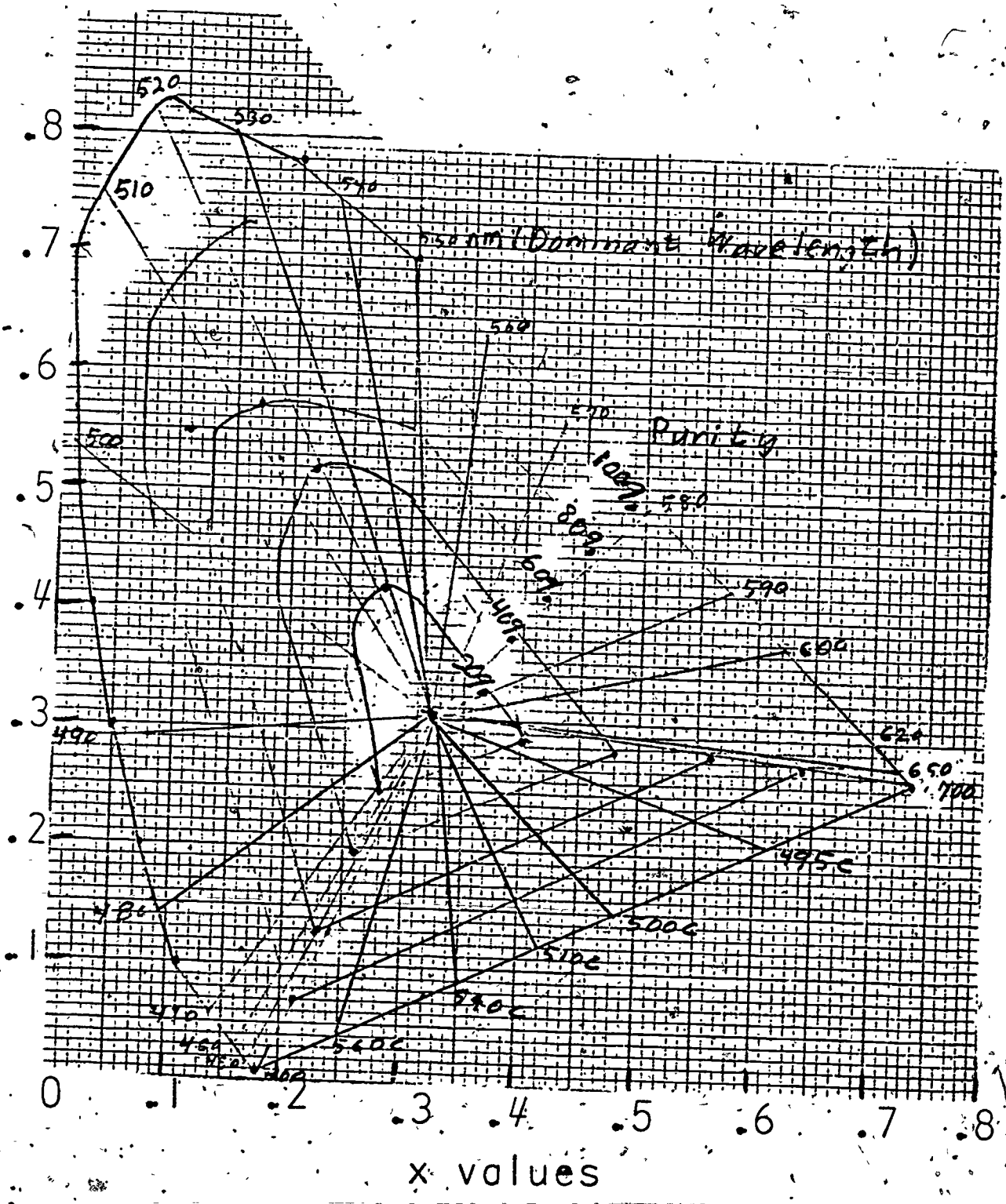
Selected Ordinates for Spectrophotometric Color Determination

ordinate number	Wavelength (nm)		
	X	Y	Z
1	435.5	489.5	422.2
2	461.2	515.2	432.0
3	544.3	529.8	438.6
4	564.1	541.4	444.4
5	577.4	551.8	450.1
6	588.7	561.9	455.9
7	599.6	572.5	462.0
8	610.9	584.8	468.7
9	624.2	600.8	477.7
10	645.9	627.3	495.2
Factor	0.09806	0.1000	0.11814

$$x = \frac{X}{X+Y+Z}$$

$$y = \frac{Y}{X+Y+Z}$$

TRANSPARENCY C09
Chromaticity Diagram



TRANSPARENCY C010

Condensed Spectrophotometer Operating instructions

1. Turn on- warmup 15 min.
   knob left front

2. Set zero
   knob left front

3. Set wavelength
   knob top right

4. Insert blank
   sample compartment

5. Set 100% T - full scale
   knob right front

6. Insert unknown
   sample compartment

7. Read absorbance or %T