

# The Sphere Photometer

## A New Instrument for the Measurement of Color and Turbidity in Solutions of White Granulated Sugars.

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### Introduction

Solutions of pure sucrose appear to the human eye as water-white and brilliantly-clear fluids. In the manufacture of white granulated sugars, the industry aims at making a product which is essentially pure sucrose. The appearance of slight coloration and haze, common to solutions of all granulated sugars, is indicative of minute traces of impurities in the product. The accurate measurement of color and turbidity in sugar solutions has been of great concern to the industry, and a variety of methods and instruments were developed and evaluated over the years. The "International Commission for Uniform Methods of Sugar Analysis" (ICUMSA) maintains standing committees on the subject (Subject 13).

Measurements of color and turbidity have a two-fold purpose: a) in process control, color and turbidity are considered indicative of the degree of decolorization and filtration achieved; b) in marketing, color and turbidity are considered to be expressive of the visual appearance of the product.

### Color

Measurements of light absorption by the solution are the basis for the characterization of sugar color. The term "color" is used here loosely, since it is used in the industry to express a) the quantity of impurities and b) the visual appearance to the human eye. Color is determined customarily by transmittancy measurements in the blue region of the spectrum. Most methods, recognized by ICUMSA and used in the industry, specify a wavelength of 420 m $\mu$ . The "color" is then expressed by an absorption index.

The colorants in sugar solutions absorb radiant energy more strongly in the blue region of the spectrum than in the red region, as shown in Figures 1 and 2. The shapes of the absorbancy curves vary significantly and it may be assumed that the variations are due to differences in raw material, processing, and pH of the solutions.

While the area under each of the curves may be a measure of the quantity of colorant in the solution, the absorbancy at a given wavelength is not necessarily a precise measure of this

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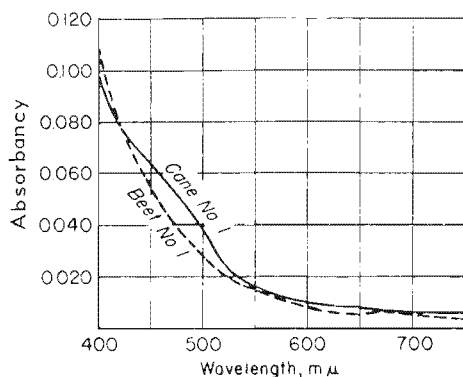


Figure 1.

Figure 2.

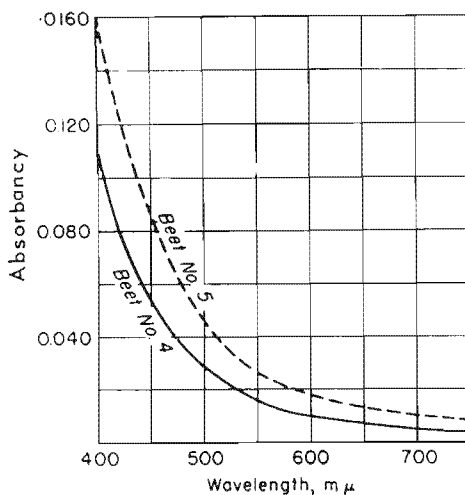


Figure 1 and Figure 2.—Absorbances of granulated sugar solutions, filtered, from 400 to 750  $m\mu$ . Reference standard: Clear, colorless sugar solution, 50 rds. solutions, 5-cm cell.

quantity. The absorbance has, however, great utility in the evaluation of the decolorization processes.

The absorption index is also regarded as being a measure of "color" in terms of visual appearance. This is erroneous since visual color perception is a psychophysical process and may be expressed quantitatively only on a three-dimensional scale, such as the CIE tristimulus coordinates [1]<sup>2</sup> and related systems. It has been shown, however, that the absorbance of sugar solutions at 420  $m\mu$  correlates well with visual rankings [2].

<sup>2</sup>Numbers in brackets refer to literature cited.

### Turbidity

In science the word turbidity has a very definite optical meaning. The general term for the phenomenon is light scattering, which the human eye perceives as a haze. It is the result of refractive index gradients in the sugar solution. While insoluble material in sugar solutions contributes the larger part of optical turbidity, the use of the word turbidity to denote a quantity of insoluble material had led to confusion. It is erroneous to assume that the colorants in sugar solutions only absorb light and that the suspensoids only scatter light. It is now considered most likely that dissolved absorbing molecules also scatter light and that scattering suspensoids also absorb light. Color and turbidity, when evaluated by optical means, are optical quantities which cannot be accurately separated by mechanical means, such as filtration or centrifuging.

Rieger and Carpenter [3] investigated the scattering of light by sugar solutions and give the following definitions:

"Turbidity is the amount of light scattered per unit path length as defined by the equation:

$$-\ln T_s = \tau b, \quad (1)$$

wherein  $T_s$  is the transmittancy of the solution,  $b$  is the path length in cm, and  $\tau$  is the turbidity in  $\text{cm}^{-1}$ . The equation applies only to systems which scatter light without absorption.

"In systems which absorb light without scattering:

$$\frac{-\log T_s}{bc} = a \quad (2)$$

wherein  $a$  is the absorption index, and  $c$  is the sugar concentration in grams per milliliter.

"In systems which absorb and scatter light:

$$\frac{-\log T_s}{bc} = a^* \quad (3)$$

wherein  $a^*$  is the attenuation index.

"Equation (1) for scattering without absorption may be written in the same form as equations (2) and (3), to define the scattering index  $s$ , as follows:

$$\frac{-\log T_s}{bc} = \frac{\tau}{2.303c} = s \quad (4)$$

"In sugar solutions, which both absorb and scatter light, the attenuation is assumed to equal the sum of absorption and scattering. In terms of the indices, one writes:

$$a^* = a + s. \quad (5)$$

(The indices of white sugar solutions are small. To eliminate fractions, we write: 1,000  $a$  = Color Index, and 1,000  $s$  = Turbidity Index.)

"In all of the preceding equations, the turbidity is expressed in terms of light lost from the transmitted beam. However, turbidity may also be evaluated by a direct measurement of all light scattered in all directions:

$$\tau = 2\pi \int_0^\pi R_\Theta \sin \Theta \, d\Theta, \quad (6)$$

wherein  $\Theta$  is the angle of observation and  $R_\Theta$  is the Rayleigh ratio, expressed as:

$$R_\Theta = \frac{i_\Theta r^2}{I_0 V} \quad (7)$$

wherein  $r$  is the distance between the scattering volume,  $V$ , and the observer,  $i_\Theta$  is the intensity of the scattered light, and  $I_0$  is the irradiance of the incident light."

Measurements of  $i_\Theta$  can be made on special photometers [4] but the complete procedure is too time-consuming for routine use. An abbreviated procedure was developed by Rieger and Carpenter [3], but the necessary instruments are quite expensive.

Photometers and colorimeters using transmission measurements in the blue and red regions of the spectrum for the estimation of color and turbidity are not completely satisfactory. The use and calibration of these instruments are predicated on the erroneous assumption that a mechanical removal of suspensoids by filtration is suitable for the estimation of turbidity.

Since turbidity can be accurately evaluated only through measurements of light scattering, Spreckels Sugar Company undertook the development of a new photoelectric instrument for the evaluation of sugar solution color and turbidity through measurements of light transmission and scattering.

### Description of apparatus optics

The optical train of the new apparatus is shown schematically in Figure 3.

Light from the filament of the lamp LS is collimated by lens L1 and passes through filter F to lens L2, which focuses the image of the filament into aperture A1 in the wall of the integrating sphere. Lens L3 again collimates the light and lens L4 brings the image of aperture A1 to focus in aperture A2. When the exit shutter is open, essentially all light entering the sphere through aperture A1 leaves through aperture A2. The interior of the sphere, the exit shutter, and the baffle are covered with a white paint of high diffuse reflectance. When the exit shutter is closed, the light entering the sphere through A1 is directed by lenses L3 and L4 to the surface of the exit shutter and reflected

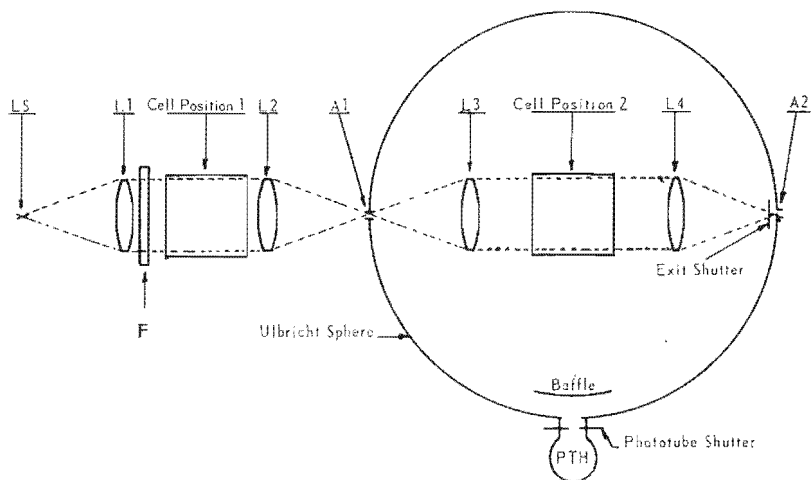


Figure 3.—Optical train, schematic.

Legend:	LS	Lamp, GE No. 1493
	L1 to L4	Lenses, achromatic, 63 mm FL, 40 mm dia.
	F	Interference filter, 420 m $\mu$ , B & L Cat. No. 33-78-42
	A1	Entrance aperture, 1.5 mm dia.
	A2	Exit aperture, 3.0 mm dia.
	PTH	Housing for IP21 phototube
	Cell	Aminco DX-5-1003, glass
	Sphere	10 in. diameter

to illuminate the interior of the sphere. After repeated reflections within the sphere, a fraction of the light illuminating the interior of the sphere enters the phototube housing PTH, where it strikes the light-sensitive cathode of the tube. The phototube is connected, through suitable electronic circuits, to calibrated potentiometers on which the light flux striking the cathode is read.

Solutions to be analyzed are placed in a cylindrical sample cell made from clear glass, having a length and diameter of 5 cm. The cell is placed into the instrument so that the collimated light beam passes axially through the cell. When the cell is in position 1, the exit shutter in the sphere is closed and the light flux reaching the phototube after reflection from the exit shutter and sphere wall is a measure of the transmittancy of the solution.

When the cell is placed in position 2, the exit shutter is opened and the light transmitted by the cell contents passes out of the sphere through aperture A2. The light scattered by the suspensoids in the sample is attenuated by the absorbers in the sample and, after reflection from the interior of the sphere,

reaches the phototube. The light flux to the phototube is a measure of the turbidity of the solution.

Since photoelectric detectors respond to radiant flux, rather than intensity, the equations presented below are in terms of flux. Designating the flux incident on the first surface of the sample as  $F_o$ , and the flux leaving the second surface of the sample as  $F_t$ , the transmittance of the sample,

$$T = F_t/F_o, \quad (8)$$

and the transmitted flux,

$$F_t = TF_o. \quad (9)$$

In the sugar industry it is customary to express the transmittancy of a sample solution relative to the transmittance of a "standard," a clear, colorless sugar solution having a sugar concentration equal to that of the sample solution. This reference solution is regarded as the solvent. The transmittancy of the sample solution,

$$T_s = T_{\text{soln}}/T_{\text{solv}}. \quad (10)$$

Expressed in terms of flux, the transmittancy of the sample solution,

$$T_s = F_{t_{\text{soln}}}/F_{t_{\text{solv}}}. \quad (11)$$

In practical applications, it is customary to standardize the photometer by placing the sample cell containing the standard solution into the instrument and adjusting it to indicate a transmittancy of unity.

Then the transmittance of the sample solution,

$$T_s = F_t. \quad (12)$$

The incident flux  $F_o$ , illuminating the colorants and turbidants in the sample solution, then equals unity, and the transmitted flux  $F_t$  expresses the transmittancy of the impurities in the sample solution. Designating the transmittancy of the absorbers of radiant energy in the samples as  $T_a$ , and the transmittancy of the scatterers of radiant energy in the samples as  $T_x$ , the following notation is applicable:

When the sample contains only absorbers, the transmitted flux

$$F_t = T_a. \quad (13)$$

When the sample contains only scatterers, the transmitted flux

$$F_t = T_x. \quad (14)$$

When both are present in the sample, the transmitted flux

$$F_t = T_a T_x. \quad (15)$$

When the sample contains only absorbers, the absorbed flux

$$F_a = 1 - T_a. \quad (16)$$

When the sample contains only scatterers, the scattered flux

$$F_x = 1 - T_x. \quad (17)$$

When both impurities are present in the sample and the transmitted and scattered beams pass through equal distances within the solution, the scattered flux

$$F_x = (1 - T_x) T_r \quad (18)$$

### Theoretical considerations in design

1. Light scattered by the suspensoids in solutions of white granulated sugars travels in a predominantly forward direction, i.e., in the direction of the transmitted beam. It is inevitable that a fraction of the scattered flux enters the detector together with the transmitted flux. The fraction  $k$  of the scattered flux entering the receiver in the transmittancy measurement is a function of the angular distribution of the scattered energy and of the angle of acceptance of the detector. The true transmittancy of a sample containing only scatterers,

$$T_x = F_t \quad (14)$$

The measured transmittancy of a sample containing only scatterers,

$$T_M = F_t + kF_x \quad (19)$$

In most currently used colorimeters, no attempt was made to exclude forward scattered light from the transmittancy measurement and the measured transmittancies of sugar solutions are significantly higher than the true transmittancies. The new photometer reduces the transmittancy error to a negligible value through the use of small apertures in the optical system.

2. In the scattering measurement according to equation (18), the length of the path traversed by the scattered beam must equal that traversed by the transmitted beam. This condition may be entirely met only in Angular Scattering Photometers [4]. In the concept of the sphere photometer it is partly met when working with solutions of white granulated sugars, since most of the scattered flux travels in a forwardly direction. The use of a clear glass cell, having equal diameter and length, facilitates inclusion of essentially all scattered energy in the measurement of scattered flux, but the condition of equal path lengths cannot be entirely fulfilled. For the measurement of scattered flux, we conceived placing the sample cell into an Ulbricht sphere (integrating sphere), and making provision for the entry of the incident flux and the exit of the transmitted flux.
3. When the measured fluxes  $F_t$  and  $F_x$  represent the functions of equations (15) and (18), transmittancies  $T_r$  and  $T_x$  may be calculated.

$$F_t = T_a T_x \quad (15)$$

$$F_x = T_a - T_a T_x \quad (18)$$

$$F_t + F_x = T_a \quad (20)$$

$$F_t/T_a = T_x \quad (21)$$

4. While considering the use of the integrating sphere in the measurement of scattered flux, it was anticipated that light reflected from the lenses and sample cell windows would augment the flux reaching the phototube. The light reflected from lens L3 and the entrance window of the sample cell is a constant fraction of the incident flux, while the light reflected from lens L4 and the exit window of the cell varies with the transmittancy of the cell contents. Designating the energy reflected by lens L4 and the exit window as  $m$ , and that reflected by lens L3 and the entrance window as  $n$ , the flux to the phototube is defined by the equation

$$F_x = (1 - T_x) T_a + m T_a + n \quad (22)$$

The evaluation of quantities  $m$  and  $n$  is discussed under "Experimental procedures."

5. Equation (22) may be written:

$$F_x = (1 + m) T_a + n - T_a T_x \quad (23)$$

Since  $F_t = T_a T_x$ , eq. (15):

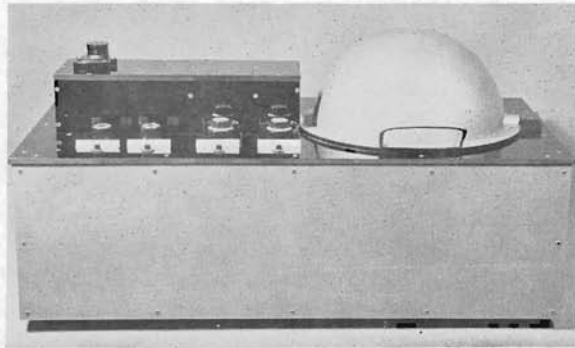
$$T_a = \frac{F_t + F_x - n}{1 + m} \quad (24)$$

### Practical considerations in design

1. Since the two separate measurements required cannot be made simultaneously, the incident flux must be held constant during the time required for the measurements. For a maximum measurement error of  $\pm 0.1\%$ , the voltage supplied to the light source, an incandescent filament lamp, is held stable within  $\pm 0.03\%$ .
2. The low light levels involved in the measurements require the use of a beam multiplier phototube. This tube has a voltage-dependent amplification. To maintain a stability of  $\pm 0.1\%$ , the applied voltage is held constant within  $\pm 0.015\%$ . The tube passes a small current when not illuminated, the dark current. Means for neutralizing this dark current are provided.
3. The two required measurements should be made with identical light sources, filters, and detectors. To meet this requirement, a single instrument with provisions for both measurement functions and using a single light source, filter, and detector is used.



Figure 4.— General appearance of the photometer.



4. Since mechanical filtration is not suitable for the separation and identification of absorbers and scatterers of radiant energy, the apparatus is adaptable to calibration by other fundamental means.
5. It is not practical to use routinely the primary standard, a 50 rds clear, colorless sugar solution, in the standardization of the instrument. Secondary standard glass plates, as used in a number of colorimeters, are subject to gradual deterioration and breakage. Manual adjustment of the transmittancy dial to the transmittancy of the secondary standard adds to manipulations required and tends to increase transmittancy errors. The new instrument uses the air path through the optics as the secondary standard and the manual standardization is automated.
6. To permit determination of flux levels to 0.1%, the apparatus employs potentiometers capable of 0.1% resolution and a null indicator of corresponding sensitivity.
7. To insure adequate photometric sensitivity, the apparatus uses a wave length of 420  $m\mu$ . The optical filter used is a Bausch and Lomb interference filter, Catalog No. 33-78-42.

#### Description of apparatus

The apparatus was designed to meet the theoretical and practical requirements outlined in the preceding sections, and to make the operation of the instrument reasonably simple and foolproof.

Figure 4 shows the general appearance of the photometer. The elevated structure on the left contains the light source, lenses L1 and L2, the filter, and supports for the sample cell in the measurement of transmitted flux (Position 1). In front of this structure are located the operating controls. The hemisphere on the right is the upper half of the integrating sphere.

Figure 5.—Sphere opened.

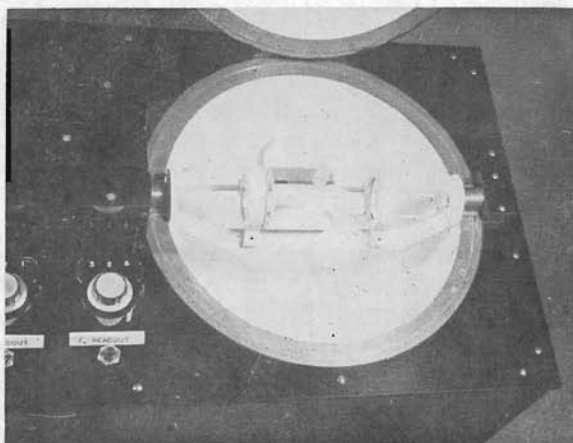


Figure 5 shows the sphere opened, with the sample cell in position for the measurement of scattered flux (Position 2). Visible at the right is the sphere exit shutter.

Figure 6—Close-up of the cell compartment and controls.

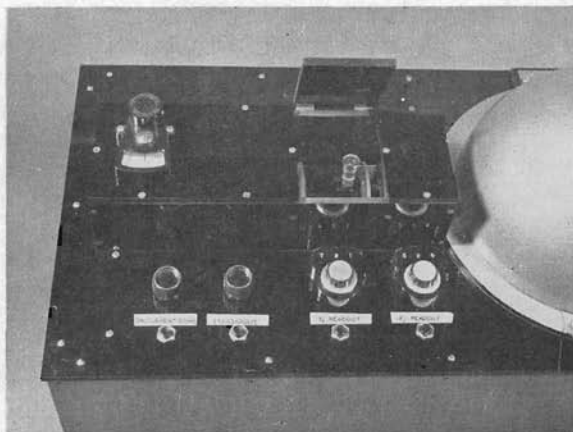


Figure 6 is a close-up of the cell compartment, operating controls, and null-indicating galvanometer. Located in front of each operating control is a pushbutton. When a pushbutton is depressed, internal circuits are switched and the shutters are actuated as needed to perform the selected function. The controls shown, from left to right, are: 1) dark current compensation, 2) standardization, 3) readout of transmitted flux,  $F_t$ , and 4) readout of scattered flux,  $F_s$ . The null indicator indicates the direction in which the selected control must be rotated to balance the corresponding circuit. When the null indicator returns to the zero position, the control function is completed.

### Experimental procedures and data

The instrument is connected to the 117 volt, 60 cycle lines through a three-wire cable, and turned on by operation of the line switch, located on the right side of the cabinet. After a warm-up period of about 5 minutes, dark-current compensation is made by manipulation of the dark-current controls. The instrument is then ready for use.

#### 1. *Standardization*

The purpose of the initial standardization procedure is the adjustment of the "Secondary Standard" to provide a reference potential related to the transmittancy of the primary standard. The reference potential permits routine standardization of the photometer, using the air in the optical path through the instrument as a secondary standard.

The primary standard, a clear, colorless 50 rds sugar solution, is prepared according to prescribed procedures. The clean sample cell, filled with this solution, is placed in position 1 in the instrument and the dark-current compensation checked. The  $F_t$  dial is set to indicate 1.000. While depressing the  $F_t$  button, the standardization control on the control panel is rotated to balance the null indicator. The sample cell is removed from the instrument and a "Secondary Standard" potentiometer inside the instrument case is adjusted, while depressing the "Standardize" button, to rebalance the null indicator. Hereafter the instrument may be standardized while the sample cell is outside the instrument, by operation of the standardizing button and control to balance the null indicator.

#### 2. *Measurement of $F_t$ and $F_x$ .*

After adjustment of the dark current and standardizing controls, the cell containing the sample is placed in position 1 in the instrument. The  $F_t$  button is depressed and the  $F_t$  control is rotated to balance the null indicator. The transmitted flux of the sample is now indicated on the digital display of the  $F_t$  control.

The sample cell is next placed in position 2 inside the sphere and the sphere is closed. The  $F_x$  button is depressed and the  $F_x$  control is rotated to balance the null indicator. The scattered flux is now indicated on the digital display of the  $F_x$  control. Flux values are indicated to three decimal places.

$F_t$  and  $F_x$  values were determined on the primary standard and on distilled water which had been filtered through a thin layer of Darco on a  $45\text{ m}\mu$  Millipore membrane for the removal of suspended solids. The values obtained are listed in Table I. The data indicate that the distilled water is suitable for use as a primary standard in the photometer at a wave length of  $420\text{ m}\mu$ .

Table 1.—Evaluation of Standards.

Sample	$F_t$	$F_s$
Primary Std.	1.000	.257
Dist. Water	1.000	.257

The scattered flux indicated is due to radiant energy reflected by the optical components in the sphere, since the primary standard is considered turbidity-free. Since the distilled water and the primary standard display identical optical properties at 420  $m\mu$ , the distilled water may be conveniently used in the preparation of artificial absorbing solutions and scattering suspensions necessary for the evaluation of the photometer.

### 3. *Photometer performance with turbid suspensions.*

Suspensions of Dicalite fines in water scatter light in a manner which closely approximates the scattering of light by the natural suspensoids in solutions of white granulated sugars. For the investigation of the relationship of transmitted and scattered flux in the photometer, such suspensions are well suited. The suspensions are prepared by dispersion of about 2 tablespoons of Dicalite in a one-liter graduate filled with distilled water. The suspension is left to settle about 24 hours. After removal of the upper 100 ml by syphoning, the following 100 ml are transferred to an Erlenmeyer flask and diluted to approximately 1 liter. The dilute suspension can be used for several days when mildly agitated by a magnetic stirrer.

Transmitted and scattered flux were measured on the photometer, on the distilled water and five suspensions of Dicalite fines are listed in Table 2.

Table 2.—Dicalite Suspensions.

Sample	$F_t$	$F_s$ (y)	$1-F_t$ (x)
Water	1.000	.257	0
Susp. 1	.986	.275	.014
Susp. 2	.963	.304	.037
Susp. 3	.895	.390	.105
Susp. 4	.753	.571	.247
Susp. 5	.548	.831	.452

From the data, we may calculate the relationship of  $F_t$  and  $F_s$ :

$$\begin{aligned}
 N &= 6 & \Sigma x &= .855 & \Sigma y &= 2.628 \\
 & & \Sigma x^2 &= .2779 & \Sigma xy &= .5727 \\
 & & & & y_c &= .257 + 1.270 x
 \end{aligned}$$

Thus the scattered flux  $F_s = .257 + 1.270 (1-F_t)$ . A plot of the data, Figure 7, shows that the scattered flux is a linear function of the transmitted flux. The constant .257 is the flux contributed by reflections from the optical components in the sphere.

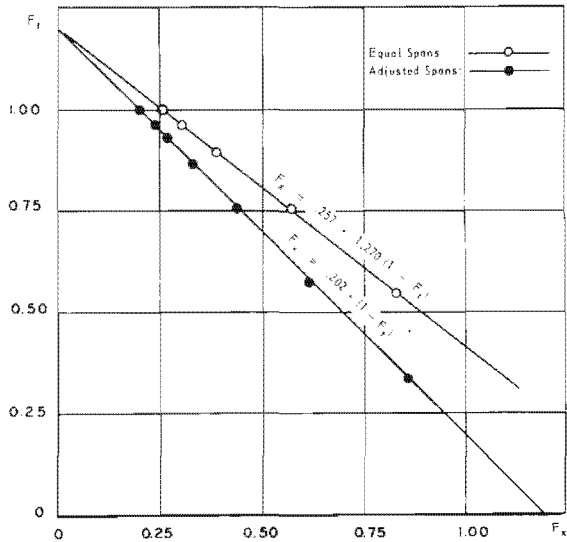


Figure 7.—Dicalite suspensions.

The factor 1.270 indicates that the flux incident on the sample in position 2 is 1.270 times the flux incident on the sample in position 1. While it is not convenient to adjust the two incident fluxes to equality, the factor 1.270 may be readily eliminated by making the span of the  $F_x$  potentiometer 1.270 times the span of the  $F_t$  potentiometer.

$$\text{Then: } F_x = .202 + (1 - F_t) \quad (25)$$

The ratio adjustment was checked with a series of Dicalite suspensions, noting the  $F_t$  and  $F_x$  values for each dilution. The data are plotted on Figure 7, showing that the scattered flux,  $F_x = .202 + (1 - F_t)$ , as calculated.

#### 4. *Photometer performance with dye solutions.*

The effect of absorbers on the measured flux  $F_x$  was investigated, using dilutions of a dye which does not contribute measurable scattering. A stock solution of a red dye was prepared in distilled water and filtered through a  $0.45 \mu$  Millipore membrane. Transmitted and scattered flux were measured on a series of dilutions as presented in Table 3.

The data were plotted and are presented in Figure 8. It is evident that for the transmittancy range of 1.000 to 0.575, flux  $F_x$  is a linear function of the transmittancy, and that the relationship may be expressed by the equation:

$$F_x = .038 + 0.162 F_t \quad (26)$$

Since a transmittancy of .575 is indicative of a color index of 78, when using a 50 rds solution in a 5 cm cell, the equation is usable

Table 3.—Dye Solutions.

Sample	$F_i$	$F_s$
Water	1.000	.200
Dye 1	.928	.188
Dye 2	.863	.177
Dye 3	.751	.158
Dye 4	.575	.131
Dye 5	.346	.103
Dye 6	.133	.078
Dye 7	.027	.068

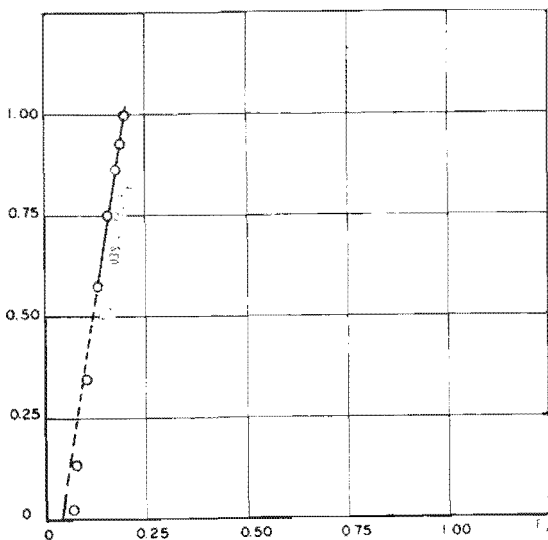


Figure 8.—Dye solutions.

for white granulated and darker sugars. It may be assumed that the constant .038 represents the radiant energy reflected by lens L3 and the entrance window of the sample cell, and the factor 0.162 represents the radiant energy reflected by lens L4. An examination of the sphere optics, Figure 3, indicates that this assumption is reasonable. The flux incident on lens L3 and the entrance window of the sample cell in position 2 is constant. When the cell contents absorb radiant energy, the flux incident on L4 is attenuated and the fraction reflected by the lens is attenuated to the same degree.

### 5. Development of working equation.

From the data and equations (25) and (26), derived independently for turbid solutions and absorbing solutions, the general equation for the flux  $F_s$ , incident on the phototube when the cell is filled with a solution which scatters and absorbs radiant

energy, may be developed. Assuming that the radiant flux scattered by the suspensoids in the solution traverses a path length through the solution which is substantially equal to that traversed by the transmitted beam, the flux incident on the phototube,

$$F_s = (1 - \Gamma_x)\Gamma_u + 0.162 T_n + 0.038 \quad (27)$$

$$F_x = 1.162 T_n + 0.038 - T_n T_x \quad (28)$$

We may now determine the transmittancy of the absorbers,  $T_a$ , by combining equations (15) and (28):

$$T_a = \frac{F_t + F_x - .038}{1.162} \quad (29)$$

The transmittancy of the scatterers:

$$T_x = F_t/T_n \quad (21)$$

To test the practical validity of equation (29), samples of different transmittancies were prepared from the dye stock solution. To these were added varying amounts of the Dicalite suspension while holding the dye concentration constant. The values of  $T_a$ , calculated from the measured fluxes  $F_t$  and  $F_x$  with the aid of equation (25), are listed in the body of Table 4.

Table 4.—Transmittancies of Dye Solutions with Added Dicalite.

Sample	ml Dicalite Suspension Added			
	0	10	20	40
	Transmittancy, $T_a$			
Water	1.000	1.000	.997	1.000
Dye 1	.964	.962	.963	.962
Dye 2	.929	.928	.928	.927
Dye 3	.866	.865	.863	.861
Dye 4	.752	.752	.751	.748

The data indicate that there is a slight reduction in transmittancy at the higher levels of added turbidity. This is not necessarily due to erroneous assumptions in the development of equation (25). It is quite possible that some interaction occurs between the dye and the Dicalite fines. The practical effect of the observed variations is best illustrated when the transmittancies are converted to indices. The "Color Index" =  $1,000 (-\log T_a)/bc$ , and the "Turbidity Index" =  $1,000 (-\log T_x)/bc$ , wherein  $b$  is the cell length in centimeters and  $c$  is the sugar concentration of the sample in grams per milliliter. The color indices calculated from the data are shown in Table 5.

The turbidity levels produced by addition of varying amounts of Dicalite are, in turbidity indices, approximately 5 for 10 ml, 10 for 20 ml, and 20 for 40 ml. The color and turbidity index range covered by the solutions exceeds the range found in solutions of white granulated sugars. The variations in the tabulated

Table 5.—Color Indices of Dye Solutions With Added Dicalite.

Sample	ml Dicalite Suspension Added			
	0	10	20	40
	Color Indices, 1,000 $(-\log T_a)/bc$			
Water	0	0	0.4	0
Dye 1	5.2	5.5	5.3	5.5
Dye 2	10.5	10.6	10.6	10.8
Dye 3	20.3	20.5	20.8	21.1
Dye 4	40.2	40.2	40.4	41.0

color indices are partly due to the uncertainty ( $\pm .001$ ) in each of the two flux measurements, which would produce an error in the indices of 0 to  $\pm 0.3$  index units. The data from dye 3 show the largest relative variation,  $\pm 0.4$  units on a mean of 20.7. This variation is  $\pm 2\%$  of the measured quantity and is considered acceptable.

#### 6. *Reproducibility of analyses.*

The overall performance of the instrument is a criterion of the stability of the electronic circuits and of the light source, as well as the sensitivity and resolution of the readout devices. To evaluate the performance of the instrument, about 2 liters of a 50 rds solution of a granulated sugar were prepared, and ten sets of readings were taken on the instrument. For each set of readings the sample cell was filled with fresh solution and the previous sample discarded. Data are given in Table 6.

The  $\Delta$  values listed in the table are deviations from the corresponding averages. Standard deviations were calculated from the data. For the color index,  $\sigma = 0.103$  and for the turbidity index,  $\sigma = 0.145$ .

#### 7. *Effect of sugar concentration on indices.*

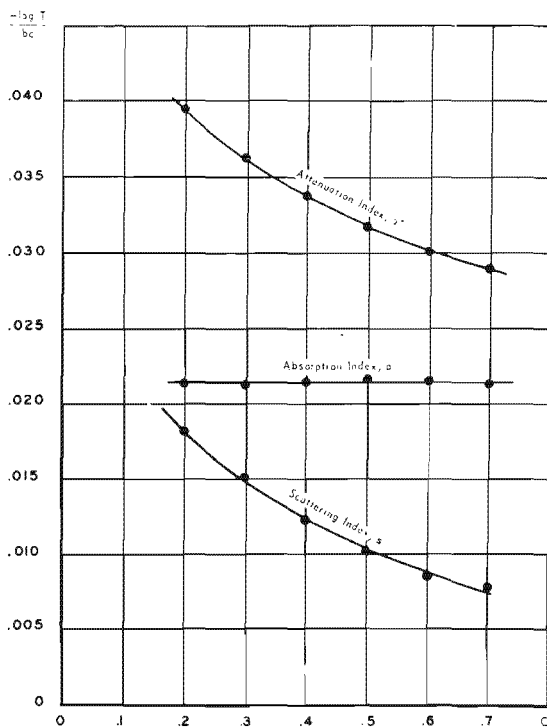
The absorbers of radiant energy in sugar solutions substantially follow Beer's law. Therefore the absorbancy index,  $(-\log T)/bc$ , is constant for any concentration of sugar and independent of the refractive index of the solution. Sugar solutions were analyzed at different concentrations in the sphere photometer to determine the attenuation, absorption, and scattering indices. The data are presented in Figure 9, which shows that the absorption index,  $a$ , is constant, while the attenuation index,  $a^*$ , and the scattering index,  $s$ , increase with decreasing sugar concentration. It is therefore necessary to report the sugar concentration at which the scattering index of a sugar solution was determined.



Table 6.—Performance Check With Sugar Solution

Test No.	F <sub>t</sub>	F <sub>s</sub>	Color Index	Turb. Index	ΔF <sub>t</sub>	ΔF <sub>s</sub>	Δ	
							Color Index	Turb. Index
1	.851	.164	18.04	4.75	-.0023	-.0015	0.13	0.17
2	.852	.163	18.04	4.58	-.0013	.0005	0.13	0.06
3	.854	.162	17.88	4.45	.0007	-.0005	-0.03	-0.07
4	.853	.163	17.88	4.58	-.0003	.0005	-0.03	0.06
5	.854	.162	17.88	4.45	.0007	-.0005	-0.03	-0.07
6	.853	.162	18.04	4.45	-.0003	-.0005	0.13	-0.07
7	.852	.164	17.88	4.75	.0017	.0015	-.03	0.17
8	.855	.162	17.71	4.45	.0017	-.0005	-0.20	-0.07
9	.855	.161	17.88	4.29	.0017	-.0015	-0.03	-0.23
10	.854	.162	17.88	4.45	.0007	-.0005	-0.03	-0.07
Avg.	.8533	.1625	17.91	4.52				

Figure 9.—Effect of sugar concentrations on indices.



### Summary

Color and turbidity in solutions of white sugars may be accurately characterized by indices of absorption and scattering. To facilitate the evaluation of the indices, a new photoelectric instrument—the Sphere Photometer—was developed for the rapid measurement of light scattering and absorption. Test data are presented which show that the measured indices are reasonably accurate estimations of the optical properties of the sample solutions.

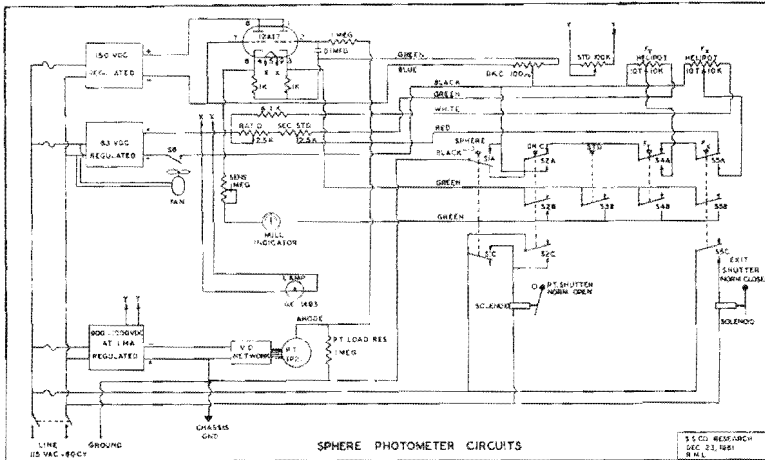
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- (3) RIEGER, C. J. and F. G. CARPENTER. 1959. Light scattering by commercial sugar solutions. J. Res. NBS. 63A (3).
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## Appendix

### Electronic Circuitry.

A schematic diagram of the sphere photometer circuits is given in Figure 10, which shows the power supplies, light source, phototube, readout circuits, function switches, shutter solenoids and potentiometers necessary for the operation of the instrument.



The 150 VDC power supply furnishes plate voltage for the 12AT7 vacuum tube, employed as a bridge-type voltmeter. Regulation for this power supply is provided by an 0A2 gas regulator tube connected across the output.

The 6.3 VDC power supply furnishes power for the light source of the photometer, a GE No. 1493 lamp, for the filament of the 12AT7, and for the potentiometers used in calibration of the photometer and in the measurement of transmitted and scattered light intensity. This power supply is completely transistorized and has a regulation of  $\pm 0.05\%$  against line voltage changes of  $\pm 10\%$ . The power supply requires cooling, which is provided by a small fan blowing air over the terminal plate. With this power supply it is necessary to apply line voltage a few seconds before the load is connected. To provide this time delay, switch S6 — a vane-actuated Microswitch — was installed in the load circuit. When the instrument is turned on by closing of the line switch, line voltage is applied to the power supply and to the cooling fan. The air blast from the fan moves the vane on switch S6 and closes the load circuit. Since the fan motor requires a few seconds to reach operating speed, the time delay necessary for the operation of the power supply is assured.

The high voltage supply furnishes operating potentials for the 1P21 beam-multiplier phototube. The highly regulated out-

put voltage may be adjusted over a range of 100 volts by a variable resistor, the standardizing control on the instrument panel.

The phototube and its associated load resistor comprise the transducer which converts luminous flux to a proportional electrical potential. The amplification of the phototube, i.e., the ratio of output voltage to luminous flux, may be controlled by adjustment of the voltage applied to the voltage divider network connected to the tube. The standardizing control on the control panel on the instrument is connected to the high-voltage supply and serves in this function. The anode end of the phototube load resistor is connected to grid 2 of the 12AT7 vacuum tube through a filter network consisting of a 1 megohm resistor and a 0.1 mfd. capacitor. The filter protects the grid circuit against alternating currents which may be picked up by the wiring. The ground end of the load resistor is connected to the negative (-) lead of the 150-volt power supply of the 12AT7 tube through one of four switch-selected potentiometers. When the potential across the selected potentiometer equals that across the phototube load resistor and is of opposite polarity, grid 2 is at the potential of the negative lead. Since grid 7 is directly connected to this lead, it is at the same potential. As a result, the currents flowing through the two cathode resistors of the 12AT7 tube are equal and the two cathodes (3 and 8) are at the same potential. Thus no current flows through the null indicator and its needle rests in the zero position, indicating balance.

The push-button actuators are connected to multi-section switches which are electrically interlocked to prevent damage to the circuitry through operator error. On the circuit diagram the switches are labeled S1 through S5. Switch sections with the suffix A select the potentiometer required for each of the functions. Switch sections with suffix B close the null-indicator circuit. Switch sections with suffix C actuate light-shutter solenoids, as required. When none of the push buttons are depressed, as during warm up or stand by of the photometer between analyses, the sample cell is outside the instrument and the light reaching the phototube is at its maximum. The potential across the load resistor is also at its maximum and, due to the position of switch section A, is opposed by the potential across the "Secondary Standard" potentiometer. The null indicator is inoperative, since switch sections B are open. When switch S3B is closed by depressing of the standardizing pushbutton, the null indicator will indicate any unbalance. Rotation of the standardization control, which adjusts the output of the high voltage supply, restores balance by matching of the potential across the load resistor to the potential across the "Secondary Standard" potentiometer.

Switch S2 performs the functions necessary for dark current compensation. When the push button is depressed, section A connects the dark current compensating potentiometer into the measuring circuit, section B closes the null indicator circuit, and section C actuates a solenoid which closes the shutter in the phototube housing. Rotation of the dark current control restores balances by adjustment of the voltage across the potentiometer.

Switch S1, connected to the lid of the photometer sphere, performs a similar function. When the lid is opened, section C energizes the solenoid and closes the shutter in the phototube housing, while section A connects the dark current potentiometer into the circuit. This interlocked function protects the phototube against excessive illumination and possible damage.

The "Dark Current" compensating potentiometer requires only infrequent adjustment because the dark current changes little during the life of the phototube.

The "Secondary Standard" potentiometer furnishes a reference potential which permits the standardization of the photometer in routine use without recourse to the primary standard.

The "Ratio" potentiometer permits the adjustment of the ratio of the potentials applied to the  $F_t$  and  $F_x$  potentiometers, i.e., the ratio of their effective spans.

The "Secondary Standard" and "Ratio" potentiometers are located inside the instrument case and are equipped with shaft locks to prevent accidental disturbance of the adjustments.

Transmittancies of sugar solutions are measured by manipulation of the  $F_t$  controls. When the push button is depressed, switch S4A connects the 10-turn Helipot potentiometer  $F_t$  to the measuring circuit and switch S4B closes the null indicator circuit. Balance is restored by rotation of the  $F_t$  control. At balance, the flux transmitted by the solution,  $F_t$ , is displayed in digital form on the control.

Scattering intensities of sugar solutions are measured by operation of the  $F_x$  controls. When the push button is depressed, switch S5A connects the 10-turn Helipot potentiometer  $F_x$  to the circuit and switch S5B closes the null indicator circuit. Switch S5C energizes a solenoid which opens the exit shutter in the sphere. When balance is restored by rotation of the  $F_x$  control, the magnitude of the scattered flux,  $F_x$ , is displayed in digital form.

A bank of four fuses for the protection of components against accidental overloads is located inside the instrument case. The instrument is connected to the electric lines through a three-conductor cable and plug, which provides grounding of the instrument case. A double-pole switch, located on the right side of the instrument, is used to turn the instrument on and off.