

Decolorizing Carbon Analyses and Tests

Describing in Detail Methods for Evaluation of the Quality and Effectiveness of Bleaching Carbons

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(In Two Parts—Part II)

THE Bureau of Standards system of color measurement is based on the following equation:

$$N = \frac{-\log_{10} T}{0.00485 Cb} \quad \text{.....(4)}$$

Where N = color of the solution
 T = transmittance of the solution (fraction of incident light transmitted) for light of a wave length 560 millimicrons.
 C = concentration of sugar in the solution expressed as grams per cubic centimeter.
 b = thickness in centimeters of layer of solution through which light passes.

The derivation of this equation may be found in "SUGAR" May 1925, pages 223 and 224, also in Technologic Paper No. 338 of the Bureau of Standards.

In Table II are given the values of—
 a) 100 T (the percent of light transmitted.)
 b) $-\log_{10} T$
 c) $\frac{-\log_{10} T}{0.00485}$ (for use in calculating the color by the Bureau of Standards' system.)
 d) $-\log_{10} T$ (The Meade-Harris color units.)

TABLE II.							
100 T	$-\log T$	$-\log T$	$-\log T$				
		0.00485	0.004355				
100 T	$-\log T$	$-\log T$	$-\log T$				
		0.00485	0.004355				
100 T	$-\log T$	$-\log T$	$-\log T$				
		0.00485	0.004355				
1	2.00000	412.37	459.3	54	.26761	55.18	61.46
2	1.69897	350.27	390.1	55	.25964	53.53	59.62
3	1.52288	314.00	349.7	56	.25181	51.92	57.83
4	1.39794	288.20	321.0	57	.24413	50.33	56.06
5	1.30103	268.23	298.8	58	.23657	48.77	54.32
6	1.22185	251.91	280.6	59	.22915	47.25	52.62
7	1.15490	238.10	265.2	60	.22185	45.74	50.94
8	1.09691	226.17	251.9	61	.21467	44.26	49.30
9	1.04576	215.60	240.2	62	.20761	42.81	47.68
10	1.00000	206.20	229.6	63	.20066	41.37	46.08
11	0.95861	197.65	220.1	64	.19382	39.96	44.51
12	0.92082	189.85	211.4	65	.18709	38.57	42.96
13	0.88606	182.70	203.5	66	.18046	37.20	41.44
14	0.85387	176.05	196.1	67	.17393	35.86	39.94
15	0.82391	169.89	189.2	68	.16749	34.53	38.46
16	0.79588	164.10	182.8	69	.16115	33.23	37.01
17	0.76955	158.66	176.7	70	.15490	31.94	35.57
18	0.74473	153.56	171.0	71	.14874	30.67	34.16

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Table II (cont'd)

72	.14267	29.41	32.76
73	.13668	28.18	31.39
74	.13077	26.96	30.03
75	.12494	25.76	28.69
76	.11919	24.57	27.37
77	.11351	23.41	26.07
78	.10791	22.25	24.76
79	.10237	21.11	23.51
80	.09691	19.98	22.25
81	.09151	18.87	21.01
82	.08619	17.77	19.79
83	.08092	16.68	18.58
84	.07572	15.61	17.39
85	.07958	14.55	16.21
86	.06550	13.50	15.04
87	.06048	12.47	13.89
88	.05552	11.45	12.77
89	.05061	10.44	11.62
90	.04576	9.43	10.51
91	.04096	8.45	9.41
92	.03621	7.47	8.32
93	.03152	6.50	7.26
94	.02687	5.54	6.17
95	.02228	4.59	5.12
96	.01773	3.66	4.07
97	.01323	2.73	3.04
98	.00877	1.81	2.01
99	.00436	.899	1.00
100

The value of T may be determined by the Hess-Ives Tintphotometer or by the use of a spectrophotometer such as the Keuffel and Esser Color Analyzer. The color of a solution as measured by a Stammer Colorimeter may also be used in the adsorption equation.

Formerly the Hess-Ives Tintphotometer has been equipped with red, green and blue violet color filters. Now it is possible to obtain a color screen which transmits light of a wave length of 560 millimicrons. This is the wave length recommended by the Bureau of Standards for the measurement of the color of sugar solutions. The Hess-Ives Tintphotometer can be purchased equipped with a $-\log T$ scale, but if one wishes to convert the reading to the color units ordinarily used, he must refer to a table, so it is just as well to purchase one with a scale reading from zero for total darkness to 100 for the full transmission of the light.

Before the color of a solution is read in one of these instruments the solution should be filtered until sparkling clear and entirely free from suspended matter. This is important, as suspended particles which contribute nothing to the actual color of the solution cut down the amount of light transmitted by it.

In taking a reading of the color of the solution in any of these instruments, the fields should be balanced by first approaching the balance from one side and taking a reading of the scale when the fields appear equal, then approaching the balance from the other side

and taking a reading when the fields again appear equal. The mean of these readings is taken as the scale reading for the solution being tested. This is done because there will be an interval of from one to five scale divisions, (depending on the transmittancy of the solution), during which there will be no appreciable change in the appearance of the fields of the instruments.

To determine the adsorption isotherm of a carbon when used with a given liquid one takes several equal portions of the liquid and treats all but one of these with increasing amounts of the carbon. The portion not given a carbon treatment is filtered and used to determine the original color of the solution. The color of each of the portions of the liquid which are treated with the carbon is measured. The "color units" removed from each of these portions, per gram of carbon used, is calculated, and the logarithm of this quotient is plotted against the logarithm of the color remaining in the solution after the carbon treatment. Instead of looking up and plotting the logarithms of these numbers, it is more convenient to use the so-called double logarithmic paper. This paper is co-ordinate paper which is graduated along each axis with a logarithmic instead of the usual uniform scale.

It is well to determine at least four points on the isotherm. As an example, let us assume that we wish to determine the adsorption isotherm of a carbon using a molasses solution.

Into each of five 250 cc. Erlenmeyer flasks measure 150 cc. of the molasses solution. Place these in a water bath and heat them to 90° C.

Take five strips of white glazed paper, each about 2 inches wide and 3 inches long. On each of these strips place one gram of Filtercel. (It is well to test the batch of Filtercel for acidity or alkalinity and if it is not neutral in its reaction to water, to either wash it until neutral or to obtain Filtercel which is neutral.) Weigh out four portions of carbon, using for instance, 0.1, 0.2, 0.4 and 0.7 grams, weighing to the nearest milligram. In weighing out these portions of carbon, one should either have the dried carbon in a stoppered weighing bottle, and in removing each portion, make no attempt to measure out exactly the amount desired, or else one should use carbon which has been allowed to come to equilibrium with the water vapor in the air, and then determine the moisture of the carbon and from this data calculate the amount of dry carbon added to each flask. Place the portions of carbon on the Filtercel on each of four of the strips of glazed paper. Add the Filtercel alone to one of the flasks,

and the Filtercel-carbon mixtures to the other four flasks. This can be easily done by slightly bending the glazed paper and allowing the mass of carbon and Filtercel to slide from the paper into the flask.

Continue heating the flasks for ten minutes after the addition of the carbon and Filtercel, shaking the flasks continuously, in order to keep the carbon thoroughly mixed with the liquid. Remove the flasks from the water bath and filter the carbon from the solutions, using ordinary 60° funnels fitted with filter paper. If the first part of the filtrate is cloudy, return it to the funnel. For color measurement, use only filtrate which is sparkling clear. Cool the filtrates to room temperature and measure the color of each. In measuring the color of the solution, choose a cell of such size that the scale readings on the Tintphotometer are not less than 10 or more than 90.

Convert the color units observed to the basis of a layer of solution one centimeter thick. This is done by dividing the observed color of the solution by the thickness in centimeters, of the layer of solution through which the light passes. Calculate the color units removed per gram of carbon and plot the isotherms.

The following example illustrates the method of recording the data and calculating the results:

TABLE III.

Grams of Carbon Used	Hess-Scale Reading	Depth of Solution Measured	Color Units for Depth Measured	Color Units per cm. Depth	Color Units Removed	Color Units Removed per gm. Carbon X/M
M.				C.	X	
0	38.7	3 cm.	85	28.3	-----	73
0.1	49.5	3 "	63	21.0	7.3	73
0.2	59	3 "	47.25	15.75	12.55	62.8
0.4	59	6 "	47.25	7.87	20.43	51.1
0.7	78	6 "	22.25	3.71	24.59	35.1

Figure II. shows C vs. X/M plotted on logarithmic paper.

The value of K. in Freundlich's equation is the value of X/M where C = 1.

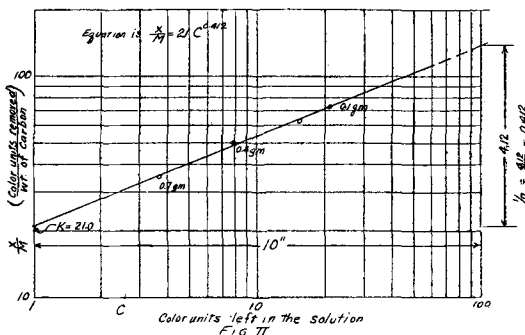
1/n is the slope of the isotherm.

This is also illustrated in Figure II.

We strongly recommend the use of isotherms in evaluating decolorizing carbons. By their use one can predict the action of a carbon on a solution with the minimum number of tests. By their use the comparison of two carbons can be readily made and recorded. The interpretation of the isotherms is easy.

Suppose we have three carbons which give the isotherms shown in Figure III. We can see at a glance that less of Carbon A. than of Carbons B. and C. will be required to decolorize the solution to any desired color within the range shown. Curves for Carbons B. and C. intersect. We know that if the final color de-

sired lies to the left of the point of intersection, Carbon C. is more efficient than B.—if to the right, B. is more efficient than C. At the point of intersection, their efficiencies will be equal. If, therefore, we are choosing between B. and C. and all their other properties are equal, and the prices are the same, we would choose C. if



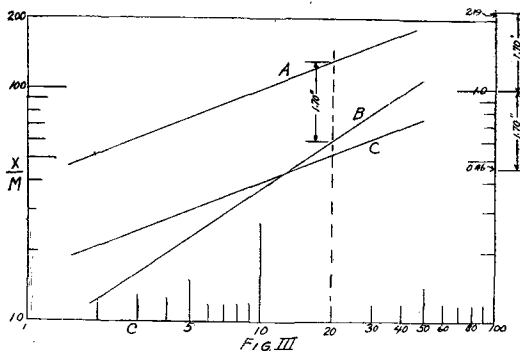
the solution is to be highly decolorized in one treatment, and B. if counter current decolorization is to be used.

The relative amounts of two carbons which are required for decolorizing the solution to any given color can readily be calculated.

At the desired final color C. we read the values of X/M for each of the carbons. Suppose these carbons are a. and b., then—

$$\frac{M}{a} = \frac{X/M}{b}$$

A still simpler method is to measure the distance between X/Ma and X/Mb on the plot and transfer this distance to the scale at the side of the paper. The relative efficiencies can then be read at a glance.



For example, suppose we wish to find the relative amounts of Carbons A. and B. (Fig. III) which will decolorized the solution in question, to a final color of 20. At 20 we find that the curves are 1.70 inches apart. We transfer this distance to the right hand scale

as indicated, and find out that to obtain this decolorization one will have to use 2.19 times as much of B. as of A., or conversely,—0.46 as much of A. as of B.

One of the advantages of logarithmic paper is that, on it, slide rule operations can be performed with a pair of dividers.

If the constants K and $1/n$ for the two carbons are known, the relative efficiencies can be calculated by the following equation:—

$$\frac{M_a}{M_b} = \frac{K_b}{K_a} C_b^{1/n_b - 1/n_a}$$

If the two isotherms are parallel $1/n_a$ equals $1/n_b$ and we have from the preceding equation:—

$$\frac{M_a}{M_b} = \frac{K_b}{K_a}$$

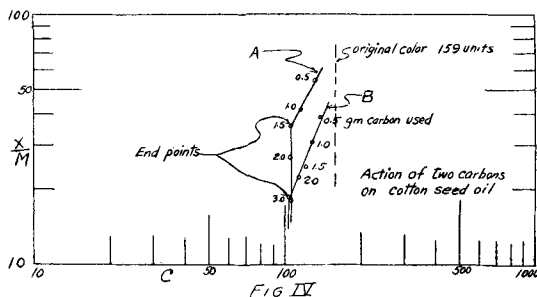
Therefore, if two carbons give parallel isotherms their relative efficiency is the same for all degrees of decolorization.

Merely grinding a carbon to a smaller particle size raises the isotherm parallel to itself. Several so-called "high power" European decolorizing carbons are made with as much as 90 to 95% passing through a 300 mesh screen. It is obvious that the high decolorizing power thus secured is at the expense of filtrability.

In plotting isotherms one may plot the log of the fraction of original color removed per gram of carbon against the log of the fraction of the original color remaining in the decolorized solution, but generally it is better to plot the log of the color units removed per gram of carbon against the log of the color units remaining in the solution.

The isotherm of a spent carbon is below that of the original carbon and usually is steeper. When studying the revivification of a spent carbon, the isotherm plot enables one to decide easily which of several methods of revivification brings the carbon back most nearly to its original condition.

In decolorizing vegetable oils, the isotherm is straight for a while, then suddenly bends vertically downward toward the log C axis. The point at which this turn takes place is called the "end point" for that carbon. No more color can be removed from that oil by the carbon in question without the use of excessive amounts of carbon. On a given oil different carbons give different "end points." It is thus possible, by means of the isotherms, to determine the carbon most suitable for decolorizing the oil.



The reason for this change in the direction of the isotherm has not as yet been determined.

In actual practice the decolorization of vegetable oils is generally done by using a mixture of fullers' earth and decolorizing carbon. The ratio of earth to carbon varies with the oil and kinds of earth and carbon used. Generally, the earth-carbon ratio is from 90/10 to 98/2 although ratios as high as 50/50 have been used.

Figure IV shows the isotherms of two carbons on cotton seed oil (C.S.O.). The end point for Carbon A. is reached when 1.5 grams of carbon have been used. The end point for Carbon B. is at 3 grams.

If a user of carbons had been accustomed to Carbon B. and tested Carbon A. by using 3 grams of it, he would obtain the same degree of decolorization and think them of the same decolorizing ability. Actually he could have decolorized his oil just as effectively with 1.5 grams of Carbon A.

The scale of color units used in Figure IV is not that used in Fig. II.

Bibliography

- Meade-Harris Color Units*.—*Industrial & Engineering Chemistry*, Vol. 12, p. 687 (1920).
- Evaluation of Carbons*.—*Chemical & Metallurgical Engineering*, 28, 541 (1923).
Industrial & Engineering Chemistry, 15, 784 (1923).
Industrial & Engineering Chemistry, 16, 498 (1924).
"Colloid and Capillary Chemistry," by H. Freundlich. Translated by H. S. Hatfield. Published by Methuen and Co. Ltd. London. This book is a translation of Freundlich's "Kolloidchemie" and is an excellent textbook on adsorption. The solid-liquid interface is discussed on pages 154 to 239.
- Transmission for Various Wave Lengths of Ives Colorimeter Filters*.
Journal of the Optical Society of America and Review of Scientific Instruments. Vol. VI. No. 6, page 568, August, 1922.
- Conversion of Stammer Color Units to Units Depending on the Extinction Coefficient of the Solution*.
See article entitled—"Die Stammersche Farbmessung" by Dr. Freda Hoffman. *Zeit. d. Ver. Deut. Zuckerind.*, Vol. 76, pages 153-161 (1926).
- The Lovibond Color System*.
K. S. Gibson and F. K. Harris, Bureau of Standards Scientific Paper No. 547. Price 15c obtainable from the Superintendent of Documents, Washington, D. C.
- Visibility of Radiant Energy*.
Gibson and Tyndall. Bureau of Standards Scientific Paper No. 475, price 15c.
- Color in the Sugar Industry*.
Peters and Phelps. Bureau of Standards Technologic Paper No. 338, price 20c.
- See also "SUGAR," May, 1925, p. 223-224.