# THE RESTORATION OF SOLARIZED ULTRA-VIOLET 1 TRANSMITTING GLASSES BY HEAT TREATMENT

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#### ABSTRACT

In this paper data are given which show the relation between the ultra-violet

transmissions of two commercial glasses (vita and helio glass) before and after solarization by ultra-violet radiations at ordinary temperatures and also after various heat treatments in the range 200° to 600° C.

It is shown that the highest ultra-violet transmission found for these glasses was obtained after heat treatments at temperatures in the annealing range; that is, somewhat above 500° C. Treatments at higher temperatures caused deformations and company the statement while these at leaver temperatures were less than a statement to the second comments are somewhat above 500° C. tions and some surface deterioration while those at lower temperatures were less effective in rejuvenating the glass, although treatments at temperatures as low as 300° C. often approximately restored the transmissivity to its initial value on receipt of the glass.

Visible colorations, which on artificial solarization accompany the decrease in transmission of the shorter visible rays, apparently disappeared completely upon relatively short heat treatments at temperatures no higher than 200° or 300° C. The visible thermoluminescence accompanying this disappearance of the coloration and apparently persisting only during the time required for the restoration of the transmission near the visible spectrum was found to grow more intense and to continue for correspondingly shorter intervals as the temperature of heat treatment was raised.

Even after the visible coloration and thermoluminescence had disappeared as a result of heating at these low temperatures, the complete restoration of the transmission for the shortest wave lengths transmitted by these glasses required

additional heat treatments near 500° C.

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## I. INTRODUCTION

During the last 10 years considerable activity in the production of glasses which to some extent replace fused and crystalline quartz for the transmission of ultra-violet radiations has been evident.

<sup>&</sup>lt;sup>1</sup> A report on the preliminary results of this investigation was read at the meeting of the American Ceramic Society held at Atlantic City in February, 1928.

activity has been encouraged chiefly by the demand for relatively large quantities of window glasses in hospital solariums, greenhouses, etc., where the need of glasses transmitting the "biologically active" ultra-violet rays 2 whose frequencies are just above those transmitted

by ordinary window glass has long been felt.3

Prior to 1910 various glasses transmitting ultra-violet radiation of the relatively short wave lengths absorbed by ordinary glass were produced and were for certain laboratory purposes available for replacing both fused and crystalline quartz which are still quite generally used in most laboratory experiments involving ultraviolet radiation. Generally these substitute glasses were expensive and unsuited for quantity production. Moreover, if they were not actually unstable, they usually lacked the resistance to weathering, etc.,<sup>5</sup> required of window glasses. Since that time, and especially since 1920, numerous glasses have been developed and tested <sup>6</sup> which transmit a fair percentage of ultra-violet radiation in the range between 280 and 320 m $\mu$  (the approximate limits of the range containing the more effective rays from a therapeutic standpoint). Moreover, some of these glasses can be produced in quantity at relatively reasonable prices.

Although most of these new glasses when first installed seemed suitable for their intended purpose and were, in general, as resistant to weathering as could be expected, it was soon found that in many cases their transmissivity for the required ultra-violet radiation decreased rapidly on exposure to the sun's rays and even more rapidly when the source of light was relatively rich in ultra-violet. decrease in transmissivity is usually accompanied by a coloration or darkening which extends throughout the whole sheet although it may be more apparent near the surface on which the light is first incident.

<sup>2</sup> The ultra-violet radiation found in sunlight, but not transmitted by ordinary window glass, is often referred to in this way. Such radiations are in many cases also termed "health rays." These and similar terms are rather indefinite and should, perhaps, be avoided, since radiations of any wave length affects living organisms in some manner and to some degree and may under certain conditions at least contribute to health, while any given wave length may at times be injurious. Moreover, the wave length or combination of wave lengths producing the maximum effect often varies considerably with the type of physiological reaction, or other effects, coming under consideration. However, the term "biologically active ultraviolet rays," although quite loose and indefinite, has been so convenient as a general designation for those invisible rays which are in and immediately below the short wave sunlight spectrum and which are mainly responsible for sunburn, etc., that it has been very generally employed.

3 Considering that it was known or suspected well before the beginning of the present century (see S. English, Glass, 5, p. 338; 1928) that ultra-violet radiation had a special significance in the medical, chemical, bactericidal, and other fields, it seems strange that the serious attempts to make sheet glasses that were transparent to this light were not made earlier. However, it does appear from certain remarks by E. Zschimmer (Zeitschrift f. Instrumentenkunde, 23, p. 360; 1903; and Physikalische Zeitschrift, 8, p. 611; 1907) that plans were being laid for developing such glasses at the approximate dates of these articles; furthermore, a statement by E. Berger (see G. Jaeckel, Glastechnische Berichte, 6, 292-293; 1928) asserts that glass transmitting ultra-violet rays was both produced in considerable quantities and used in windows prior to 1914. (See also, E. Berger, Sprechsaal, 61, p. 564; 1928.)

4 Eder u. Valenta, Denkschrift d. Wien. Akad. Wiss. (Math.-Nat. Kl.), 61, p. 285; 1894. E. Zschimmer, Physikalische Zei

Colorations 9 resulting from exposure to solar or artificial ultraviolet and to Röntgen or radium radiations had previously been observed in many glasses and other materials; and such colorations have been ascribed to various causes. Coloration produced in ordinary glass when exposed to the sun's rays has in some cases been associated with a content of manganese, selenium, etc.<sup>10</sup> In the ultra-violet transmitting glasses, however, the opinion of many investigators seems to be that the observed coloration on exposure to ultra-violet radiation is ordinarily due to a reoxidation of ferrous to ferric iron. 11 Investigations on the effect of these oxides on the transmission of glass and the fact that melting glasses in a reducing atmosphere or keeping the iron content to a minimum increases the ultra-violet transmissivity supports this opinion. 12

It is possible, however, that other factors besides a change in the oxidation of the iron, and also that other elements besides iron may

be operative in this reduction of ultra-violet transparency.

As an example of the variety of views which may be suggested by experience with similar effects in other materials, attention is drawn to one which has been advanced and which ascribes the reduction of the ultra-violet transmission to a gradual separation of finely divided sodium in the glass. Although this separation effect has been observed in some sodium compounds when they are subjected to certain radiations, its particular significance appears, however, to be very questionable 13 in the case of this aging effect in glass.

In this connection it may also be noted that samples of fully transparent crystalline quartz from certain sources appear to suffer, as a result of exposure either to ultra-violet or to cathode rays,14 no coloration, and presumably, therefore, little, if any, diminution in their power of transmission for ultra-violet rays; while, on the other hand, fused quartz supposedly from crystals of the same kind has been found to show (at least in certain cases) both coloration and diminished transmission to some degree <sup>15</sup> after such exposures. It is also reported that the purple coloration of the latter material by cathode rays is formed in spots. 16 Such results would seem to indicate that the colorations are due to some impurity which is introduced or changed during the melting of the quartz and that this impurity, which may or may not be iron, is also the cause for the "solarization" occasionally observed in the resulting glass. suggest, moreover, the possibility that the condition causing these spots is also involved in the formation of the thin filmy colorations sometimes observed in certain pieces of fused quartz which have

<sup>&</sup>lt;sup>9</sup> W. Crookes, Phil. Trans. (II), p. 170; 1879. P. and M. Curie, Comp. Rend., 129, p. 823; 1899. C. Doelter, Das Radium und die Farben (Steinkopf, Dresden); 1910. St. Meyer, Phys. Zeitschrift, 10, p. 483; 1909. S. C. Lind, J. Phys. Chem., 24, p. 437; 1920. Parmelee, Clark, and Badger, J. Soc. Glass Tech., 13, p. 279; 1929.

<sup>10</sup> S. Avery, J. Am. Chem. Soc., 27, p. 909; 1905. F. Fisher, Ber. d. Deut. Chem. Ges., 38, p. 946; 1905. Meyer and Przibram, Sitz. Ber. Akad. Wien (IIa), 121, p. 1414; 1912. A. Dauviller, Comp. Rend., 181, p. 601; 1925. F. Eckert, Zeit. Tech. Physik., 7, p. 300; 1926. Coblentz and Stair, B. S. Jour. Research, 3 (RP113), p. 629; 1929.

<sup>11</sup> Starkie and Turner, J. Soc. Glass Tech., 12, p. 306; 1928.

<sup>12</sup> Corning Glass Works, Brit. Patent 263410; July 1, 1926. Starkie and Turner, J. Soc. Glass Tech., 12, p. 27; 1928. A. Rüttenauer. (See footnote 8, p. 358.) G. Rose, Sprechsaal, 62, p. 354; 1928.

<sup>13</sup> G. Rose, Sprechsaal, 62, p. 375; 1929.

<sup>14</sup> Coolidge and Moore, J. Frank. Inst., 202, p. 724; 1926.

<sup>15</sup> See footnote 14. Coblentz and Stair. (See footnote 10.)

been heated 17 for some time at temperatures in the neighborhood of 1,000° C. Although there may be no definite relation between these two types of colorations the apparent similarity of the patterns formed certainly supports this view, since these patterns both seem to outline the shapes of the original crystal fragments. According to Coolidge and Moore, however, the cathode ray colorations disappear on strong heating; consequently the reactions causing the two effects must be different even if the basic conditions responsible for both are the same.

It is characteristic of the colorations caused in glass by various radiations that they will usually change, decrease, or completely disappear under the influence of heat; 18 and, in some cases, they change merely through exposure to radiation of a different character or frequency. 19 Generally this disappearance induced by heating is associated with a more or less brilliant thermoluminescence.20 It has been reported by various investigators that both the brilliancy of this thermoluminescence and the rate of disappearance of the coloration increase rapidly as the temperature of the restoring heat treatment is increased.<sup>21</sup> In the case of ultra-violet transmitting glasses which have been solarized by exposure to radiation of the sun or quartz mercury lamps the transmissivity for the ultra-violet is, as might be expected from the disappearance of visible coloration, more or less restored by heat treatment 22 and the rate of this restoration also increases with temperature.

The results in the preliminary report of this investigation, which was undertaken early in 1927 for the purpose of gaining some notion of the relationship between solarization, coloration, thermolumin-escence and the restoration of transmissivity by heat treatment for some of the glasses transmitting ultra-violet radiation, have already been reported.23 The conclusions at the time of making the report were that the decoloration could be practically completed and visible thermoluminescence exhausted by heat treatments at temperatures near 300° C., but that a complete restoration of the transmissivity to the possible maximum required treatments at temperatures much nearer 500° C., which was in the annealing range 24 of the glasses

In a sense this corresponds with the pervious experience of Lind 25 and others who found that the "discharge" of the brown color produced

It has been suggested to the authors of the paper cited here that the markings caused by heat treatment may arise from some change in films of carbon and silica compounds which are formed (possibly) by the action of the carbon vapor atmosphere which is undoubtedly present during melting and which supposedly permeates all of the evacuated spaces between the individual fragments of the quartz charge. Presumably, reheating the resultant glass to temperatures near 1,000° C. might cause these films to darken so that they become visible, if finely divided silicon or carbon is deposited in the glass through some reducing action during prolonged heating at these temperatures. Furthermore, such films and impurities may ultimately induce devitrification causing the original pieces in the charge to be outlined by the light colored films sometimes observed after heating certain samples of the glass for a few hours at somewhat higher temperatures.

sometimes observed after heating certain samples of the glass for a few hours at somewhat higher temperatures.

15 Meyer and Przibram. (See footnote 10, p. 359.) S. C. Lind, J. Phys. Chem., 24, p. 440; 1920. J. R. Clarke, Phil. Mag., 45, p. 735; 1923. F. Eckert, Zt. Tech. Physik, 7, p. 300; 1926.

16 F. Fisher. (See footnote 10, p. 359.) Meyer and Przibram, p. 415. (See footnote 10, p. 359.) P. L. Baley, Phys. Rev. (2), 24, p. 495; 1924. Coblentz and Stair (B. S. Tech. News Bull., No. 160, p. 80; and No. 162, p. 97; 1930) report that in some cases at least the transmission of a solarized glass is increased when the glass is exposed to ultra-violet radiation of a longer wave length than that causing solarization.

20 See reference under footnotes 18 and 21.

21 Nyswander and Lind, J. Opt. Soc. Am., 13, p. 660; 1926.

22 Coblentz and Stair. (See footnote 10, p. 359.) H. P. Hood. (See footnote 24.)

23 Tool and Stair, unpublished Progress Report read at the Annual Meeting Am. Ceram. Soc., Atlantic City, Feb. 5 to 11, 1928.

24 This conclusion agrees with results obtained by Reinhardt and Schreiner, J. Phys. Chem., 32 (2), p. 1886; 1928. See also H. P. Hood (Corning Glass Works) Brit. Patent No. 298908, Oct. 15, 1927.

25 S. C. Lind (See footnote 18.)

in a glass by radium radiation was accomplished by gentle heating and was accompanied by thermoluminescence, but that the discharge of the violet color caused by the same radiation did not take place until a tempreature of 500° C. was reached, which was near the softening point of the glass. Moreover, after the thermoluminescence was exhausted at 200° C. it did not appear again at higher temperatures between 200° and 500° C. On the other hand, in the year 1928 Shrum, Patten, and Smith <sup>26</sup> reported that the thermoluminescence could be not only exhausted at temperatures near 300° C., but further that the effect of solarization also disappears completely at the same time.

The transmission results given in the above-mentioned preliminary report are shown in Figure 5 (circles), and indicate the degree of restoration then attained by heat treating at different temperatures certain solarized samples of one of the ultra-violet transmitting window glasses (vita glass) on the market at that time. Since the conclusions reached in this report were based mainly on the results obtained by the use of only two wave lengths, namely 280 m $\mu$  and 313 m $\mu$ , and since it was apparent that results at other wave lengths might have additional interest, it was decided to repeat the work on a more elaborate scale using five wave lengths as follows: 280 m $\mu$ , 313 m $\mu$ , 334 m $\mu$ , 365 m $\mu$ , and 405 m $\mu$ . The method employed and the results obtained are discussed in the following paragraphs:

## II. GLASSES TESTED

#### 1. SAMPLES USED FOR TRANSMISSION TESTS

Two window glasses bearing the trade names vita and helio glass were used in this investigation. Strips were cut from panes of these glasses and divided into a number of samples from which 10 for each glass were chosen. The samples selected were those which apparently had the best surfaces and the fewest imperfections (such as striæ) although some effort was made at the same time to obtain for each glass a set of samples which would be representative of the whole length of a single strip. The dimensions were about 2 by 3 cm, while the thickness varied considerably. This variation was not so marked over any single sample but for the vita glass the thickest and thinnest samples measured 3.13 and 2.87 mm, respectively, and for the helio glass 2.39 and 2.05 mm.

#### 2. APPROXIMATE ANNEALING AND SOFTENING RANGES

In order to gain some indication of the annealing and softening ranges of these glasses heating curves <sup>27</sup> were obtained. These curves are shown in Figure 1, and a description of the individual curves is given in the accompanying legend. Based on general experience with other glasses it appeared from these curves that the annealing ranges for ordinary purposes were in both cases near and somewhat above 500° C. and that considerable deformation might be expected if the glasses were heated for any extended time at 600° C.

Shrum, Patten, and Smith, Trans. Roy. Soc. Canada, (3) 22, p. 433; 1923.
 Tool and Valasek, B. S. Sci. Papers, 15, p. 537; 1920. Tool and Eichlin, J. Opt. Soc. Am., 4, p. 340; 1920.

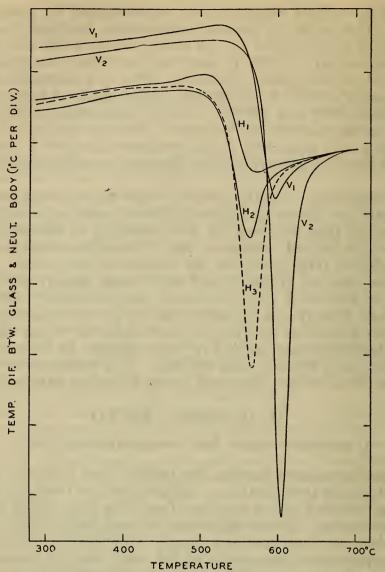


FIGURE 1.—Heating curves obtained on vita and helio glasses

Tests on the glasses in their original condition resulted in heating curves  $V_1$  and  $H_1$  for the vita and helio glass, respectively. The relatively pronounced drop of  $V_1$ , as compared to that of  $H_1$ , to a minimum below the final level of the curves indicates that the original vita glass was annealed to a somewhat greater degree than the original helio glass. This indication was supported to some extent by the character of the resistance of the panes to the glass cutter; that is, the helio glass pane appeared to have a thin surface layer which was still under a relatively high compression.

Curves  $V_2$  and  $H_2$  were obtained on samples of these glasses after they had been heat treated together. In this treatment the samples were heated to 530° C. and cooled according to a schedule which would approximate a steadily decreasing rate requiring 12 days to reach 490° C., the point at which they were removed from the furnace to cool rapidly to room temperature. This treatment increased the degree of annealing over that of the original in the case of both glasses, but was far more efficient for the vita than for the helio glass. These conclusions are reached in view of the increases in the heat absorption effects and the relatively large effect shown by curve  $V_2$ . This difference between the heat absorption effects of curves  $V_2$  and  $H_2$  is in part, at least, accounted for when it is noted that the heat absorption range, and consequently the annealing range, of the helio glass is not less than 30° below that of the vita glass.

To show more clearly that the discrepancy in the indicated degrees of annealing of the two glasses was

vita glass.

To show more clearly that the discrepancy in the indicated degrees of annealing of the two glasses was probably due to the fact that 490° C. is not a comparable point in the annealing range of these glasses and to show also that it may be reduced by continuing the treatment of the helio glass to a lower temperature, this glass was reheated to 490° C. and cooled slowly to 470° C. before again taking it from the furnace. Curve H<sub>3</sub> was obtained after this treatment and the increase in the heat absorption effect indicates that the degree of annealing was increased. (See article entitled "Variations Caused in Heating Curves of Glass by Heat Treatment," by Tool and Eichlin, B. S. Jour. Research, 6, p. 523; 1931.) It also shows that if the two glasses were annealed to a comparable degree; that is, if they were brought to equilibrium at temperatures which were about equally distant from the temperatures determined by the minima of their respective heating curves, the heat absorption effects as measured by the magnitude of the drop below the final level of the curves would probably not be greatly different for the two glasses. glasses.

(Legend continued on p. 363.)

## III. EXPERIMENTAL PROCEDURE

## 1. SEQUENCE OF TESTS AND TREATMENTS

After the samples had been chosen the transmission of each was measured for the five wave lengths previously mentioned. All samples were then artificially solarized and the transmissions again determined. Following this the power to transmit ultra-violet radiation

was more or less restored by heat treatments.

For these heat treatments five samples of each glass were put in lot A and the rest in lot B. The samples of each lot were always treated together. Those in lot A were first treated at 200° C., and then, as indicated in Tables 3 and 4, they were retreated successively at the higher temperatures until 575° C. was reached. Those in lot B were first treated at 575° C. and were later given treatments successively at the lower temperatures indicated. Finally all samples were treated at 600° C. This treatment was given last because it was known that some deformation and possibly some injury to the surfaces would result.

After each of these heat treatments at any temperature the transmissions were determined, and the samples were then solarized again before the next heat treatment. As a check on the uniformity of the degree of solarization caused by the ultra-violet treatments the transmissions of at least two samples from each glass in a lot were determined for 313 m $\mu$  after each solarization.

#### 2. TRANSMISSION MEASUREMENTS

The transmission of the glass samples at the five wave lengths chosen were obtained radiometrically by using a quartz mercury lamp, a quartz spectrometer, and a vacuum <sup>28</sup> thermopile. An ironclad <sup>29</sup> galvanometer used in conjunction with the thermopile was adjusted to a sensitivity sufficient to obtain deflections of several centimeters for the weakest mercury emission lines used. A fluorescent screen (a coating of anthracene) on each side of the entrance slit of the thermopile furnished a visual method of calibration for the spectrometer.

Measurements at a given wave-length setting consisted in observing the galvanometer deflections with and without the sample inserted in the beam of light at the entrance slit of the spectrometer. Several readings were made for each wave length and each sample. The ratio of the readings (sample in to sample out) at any setting

gave the transmission of the glass at that wave length.

#### FIGURE 1-Continued

Coblentz, B. S. Sci. Paper No. 413, 17, p. 187; 1921.
 Coblentz, B. S. Sci. Paper No. 282, 13, p. 423; 1916.

It will be noted that the difference between the initial and final levels of these curves is in every case much greater for the vita than for the helio glass. The discrepancy between these differences indicates merely that, during the passage of the glasses through their heat absorption ranges, the change in the thermal properties of the vita glass is such that the temperature gradient from the containing walls into the sample must be increased on reaching points above the absorption range by a relatively large amount as compared to that required in the case of helio glass if the heating rate chosen as standard is to be maintained.

Although the heat absorption range, and presumably the annealing range also, of the helio glass is below that of the vita glass, it was found that on heating the former to temperatures above 650° C. it showed slightly less sintering between the particles used in the heating curve test than the latter glass under the same conditions. This indicates that the rate of softening is lower for the helio than for the vita glass and explains why the former withstood the rejuvenating treatment at 600° C. as well as the latter, although the relative thinness of the helio glass samples seemed to make deformation more probable.

In all cases the glasses were thoroughly washed with soap and water, carefully rinsed in clear water, and dried both before making transmission measurements and before placing them in the lamp box for solarization treatments.

#### 3. SOLARIZATION TREATMENTS

Upon exposure to the radiation from a quartz mercury arc lamp all glasses that have been examined decrease in ultra-violet transmission, some decreasing more than others. Certain samples of fused quartz have also been found to decrease slightly in transmission in

the shorter wave lengths (265 mµ and below).

For vita and helio glass, however, the greatest change in transmission from such an artificial solarization treatment occurs, as might be expected, in the range of wave lengths below 405 mµ since it is near this point that the region of high absorption of radiation by these glasses first becomes noticeable. In consequence, the transmission experiments of this investigation have been confined entirely to this region of rapidly increasing absorption with decreasing wave lengths

and of greatest change in transmission from solarization.

The time of exposure used for these glasses was chosen after a study of the solarization results obtained by exposing samples for different periods of time. From such results it was found that, when the samples were exposed (at a distance of 15 cm) to a 110-volt quartz mercury arc lamp operated with 70 volts across the burner, most of the change occurred in the first 3 or 4 hours and that after 5 hours any further change was very slow. This indicated that the practically flat portion of the time-solarization curves had been reached; hence a period of five hours was chosen for all the exposures in these tests. In the beginning some tests were made to determine the effect of turning the samples over once during this time in order to expose both sides directly; and while this increased the effectiveness of the treatments somewhat the difference did not appear to be enough to warrant a continuance of the practice.

On each exposure throughout these experiments, therefore, the samples were placed upon a board 15 cm beneath the lamp and, excepting the few cases noted, the marked sides were always toward this source. The distance of 15 cm was sufficient to prevent any heating which might unduly counteract <sup>30</sup> the desired effect of this

<sup>30</sup> One of the first preliminary tests concerned this possibility. A sample while being held at temperatures somewhat above 400° C, suffered no solarization when exposed to the radiation concentrated on it from a small quartz mercury lamp by a quartz lens. In reality its transmissivity increased somewhat if there was any effect.

at a lower temperature, however, a considerable increase in its transmission may be noted as the result of such a simultaneous treatment.

At somewhat lower temperatures the counteracting effect of the heating being relatively lower causes the resulting equilibrium conditions from a simultaneous solarization to yield lower transmissions; but even so the effect of temperature has considerable significance in the case of glass bulbs used in ultra-violet lamps since such bulbs are usually heated to no small degree and so continue to possess a satisfactory transparency to ultra-violet radiation.

At temperatures nearer that of the atmosphere, however, the rate of restoration by heat is apparently much less than that of the solarization received from a reasonably effective ultra-violet source by a sample which is in an intermediate condition as a result of previous treatments; consequently a solarization at

was any effect.

As a matter of fact the data available at present on these glasses indicate that the rates of restoration by heating and of solarization by ultra-violet radiation decrease, respectively, as the maximum restoration and solarization obtainable are approached and that both decrease with the temperature, the former decreasing more rapidly. At relatively high temperatures, it appears that the rate of rejuvenation from heating alone may be much greater than the rate of solarization caused by a given source while the glass is held at these temperatures. Consequently, the kinetic equilibrium established by a solarization treatment at any such temperatures (by simultaneous solarization and rejuvenation treatments) should result in an ultra-violet transmission relatively near the maximum obtainable by the heating alone; that is, the solarization of a sample previously rejuvenated in the absence of radiation will be small or negligible on being subjected to a solarization treatment at temperatures near 500° C. With a sample previously solarized at a lower temperature, however, a considerable increase in its transmission may be noted as the result of such a simultaneous treatment.

aging treatment; furthermore, since the samples were small, this distance was large enough so that their relative positions with respect to the lamp differed very little. These conditions, together with the fact that the time of exposure corresponded to a relatively flat portion of the time-solarization curve, aided in erasing and in preventing the building up of any possible differences of undue magnitude between the degrees of solarization of the different individual samples of either of the glasses.

These solarization treatments, as previously stated, always followed the transmission tests and preceded the heat treatments. The abovementioned check transmission measurements on a few samples after each solarization treatment showed that within reasonable limits these various radiation treatments always reduced the transmissions for a given wave length to practically the same minimum value.

## 4. HEAT TREATMENTS

The glasses were heat treated in an electric furnace which could be maintained at a relatively constant temperature for several days. During these treatments the samples were packed quite closely together, although the surfaces were never in contact with each other or with other materials except near the edges where the platinum or aluminum foil spacers rested. To protect the surfaces from dust, the carefully cleaned and dried samples were inclosed in a small aluminum box.

Before the treatment began the furnace was raised to, and stabilized at, the desired temperature. After introducing the samples the temperature seldom fluctuated by as much as  $\pm 5^{\circ}$  C. and the average was always very close to the desired treating temperature. The possible errors in the temperature determinations by the platinum, platinum-rhodium thermocouple employed was considered to be of about the same order as the fluctuations. It is to be noted, however, that these errors in the indicated temperatures would in most cases be of the same sign; consequently the intervals between the average temperatures of the successive treatments are probably accurate to within 5° C.

The heat treatments of the two lots, A and B, were begun at opposite limits of the treating range because it was believed that the results at any point in the series of tests might depend somewhat on the temperature of the previous treatments. As indicated in Tables 3 and 4, the periods of treatment for lot A were longer than those for

these temperatures produces a marked reduction in transmission. Within this whole range of intermediate temperatures it appears that solarization (regardless of whether equilibrium is or is not reached) may always be carried well beyond the degree of reasonable constancy after the radiation is stopped. The return, accompanied by increasing transmission, to an approximately constant condition (such conditions may fall far short of the limits approached at such temperatures in the absence of radiation) probably causes much of the brilliant phosphorescence observed at these temperatures whenever the radiation of a sensitive glass by the ultra-violet source ceases.

At relatively low temperatures, however, where the rate of solarization becomes still lower it is probably impossible to reach an equilibrium condition by a solarization treatment of any practical length; and it may even be impossible to reach those degrees of solarization which are easily obtainable at higher temperatures (somewhat above that of the atmosphere, for example). If this be the case, it should be possible by a series of ultra-violet treatments for equal times from a given source to establish the temperatures yielding optima for solarization, phosphorescence, thermoluminescence, etc.

With regard to the effect of temperature on solarization near atmospheric temperatures, Coblentz and Stair (B. S. Jour. Research, 3, p. 647; 1929) found that at a distance of 15 cm from the quartz mercury arc lamp the temperature was of the order of 70° C. and that at this temperature vita and helio glass solarized to alower transmission value than when exposed at 15° C. to the same radiation. This probably indicates that, under the conditions of their experiments, the optimum temperature rejuvenates these glasses appreciably.

B and in the cases of both A and B they were considerably longer at low temperatures. The object of these differences in the periods of treatment of the two lots was to determine whether the periods, particularly at low temperatures, were great enough to procure approximately the maximum effect at each treating temperature.

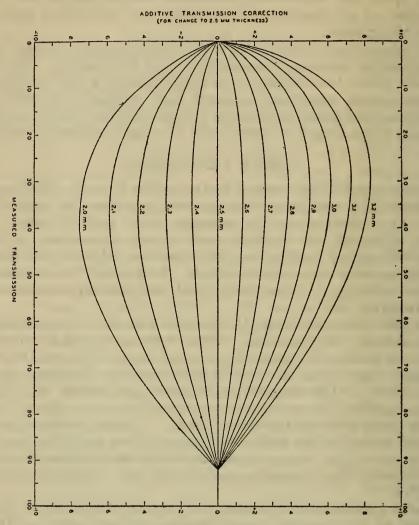


Figure 2.—Curves giving transmission corrections required because of variations in sample thickness

The equations necessary for computing the data used in the preparation of such curves may be obtained from the relations which show the variation of transmission with thickness and which are to be found in various texts and articles; for example, the article by Gibson and McNicolas in B. S. Tech. Paper, No. 119, p. 11; 1919. The above curves were prepared on the basis of a 4 per cent reflection at each surface of the specimen. At times it is necessary to allow for a larger percentage of reflection, especially near absorption regions. This may require the preparation of additional graphs, but usually sufficiently accurate adjustments can be made without this additional labor if the actual reflection percentage does not deviate too greatly from that assumed in the preparation of the curves. For a different type of correction chart see the revised edition of the Gibson and McNicolas paper cited above.

#### IV. RESULTS

#### 1. TRANSMISSION

For the purpose of easier comparison the transmission values given in Tables 1 and 2 are adjusted to a thickness of 2.5 mm, which is about the mean thickness when all of the samples of the two glasses tested are considered. (For adjustment method employed see fig. 2.) These adjusted values for all 5 wave lengths and for all 20 samples,

both before and after their first solarization, are given here in order to indicate the degree to which the results were consistent. To show something of the magnitude of the corrections required in the adjustments, the observed transmissions and the corrections for the two extreme wave lengths employed are also included in the tables.

In making these adjustments it was found that the same results for the average adjusted transmissions could be obtained by using the average observed transmissions and thickness. Further tests in applying such corrections to the individual and average results both after heat treating and after resolarizing the samples showed that sufficiently accurate adjustments could be made if the average

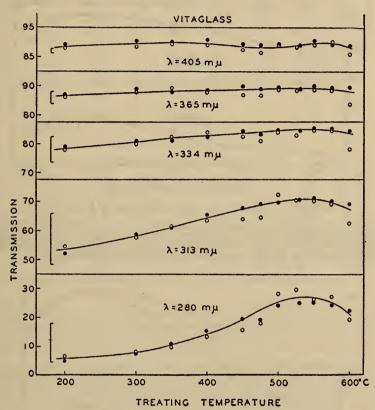


Figure 3.—Results for transmission of vita glass

The circles and large dots represent the transmission results obtained on the samples of lots A and B, respectively, after the various heat treatments, lot A being treated at increasing and B at decreasing temperatures. The upper and lower ends of the bracket at the beginning of each curve represent the transmissions of the glass when it was in the original condition and after its first solarization.

transmissions and thickness were used for each of the sets of five samples designated as lots A and B. Consequently this method of adjusting the averages was followed in obtaining the results (Tables 3 and 4) showing the effects of alternate solarization and heat treatment. These results are also graphically represented in Figures 3 and 4, while for the purpose of comparison Figure 5 reproduces on a different scale the probable curves found in Figures 3 and 4 for the wave lengths 280 m $\mu$  and 313 m $\mu$ . The circles in Figure 5 represent the preliminary transmission results on vita glass which were discussed in the progress report read at the American Ceramic Society meeting previously mentioned.

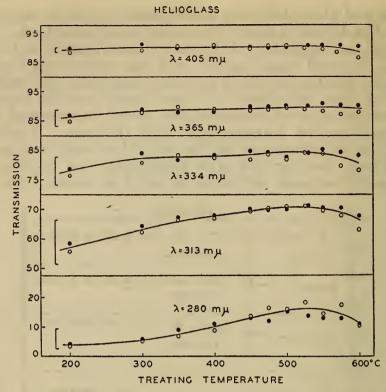


FIGURE 4.—Results for transmission of helio glass

The circles and large dots represent the transmission results obtained on the samples of lots A and B, respectively, after the various heat treatments, lot A being treated at increasing and B at decreasing temperatures. The upper and lower ends of the bracket at the beginning of each curve represent the transmission of the glass when it was in the original condition and after its first solarization.

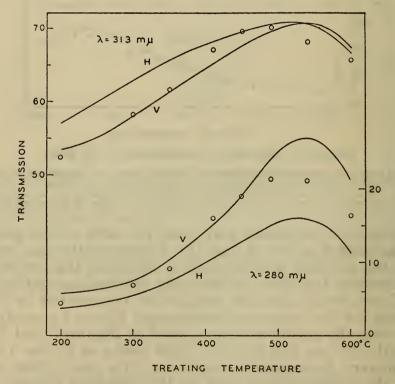


Figure 5.—Comparison of results on transmission of vita and helio glasses

Curves V and H reproduce on a different scale the trends of the curves for 280 m $\mu$  and 313 m $\mu$  shown in Figures 3 and 4, respectively. The circles represent some preliminary results for vita glass.

## Table 1.—Vita glass

### INITIAL TRANSMISSIONS 1

					Wa	ve lengtl	ns <del></del>						
Sample No.	Thick- ness	280			313	313 334		405					
		Ob- served	Correc- tion	Ad- justed	Ad- justed	Ad- justed	Ad- justed	Ob- served	Correc- tion	Ad- justed			
2	mm 3. 10 3. 12 3. 16 3. 08 3. 06 3. 02 2. 92 2. 89 2. 87 3. 03	12. 1 12. 1 11. 8 12. 3 12. 2 12. 5 12. 6 12. 9 14. 0 14. 2	5. 8 6. 0 5. 9 5. 8 5. 5 5. 2 4. 2 4. 1 3. 8	17. 9 18. 1 17. 7 18. 2 18. 0 17. 8 17. 1 18. 1 18. 0 17. 8	65. 6 67. 0 65. 3 65. 3 66. 2 65. 2 64. 8 65. 6 66. 6 65. 8	81. 8 80. 6 82. 2 82. 8 82. 8 83. 9 82. 3 81. 6 82. 7 81. 5	87. 1 89. 2 87. 8 87. 6 87. 1 88. 9 88. 3 88. 6 87. 9 88. 0	84. 4 87. 1 85. 8 86. 5 88. 3 86. 5 88. 0 87. 1 87. 5 88. 0	1. 4 1. 0 1. 2 1. 1 . 7 . 9 . 7 . 7 . 6 . 5	85. 8 88. 1 87. 0 87. 6 89. 0 87. 4 88. 7 87. 8 88. 1 88. 5			
	T	RANSM	IISSION	S AFT	ER SOI	LARIZA	TION		[				
2	3. 10 3. 12 3. 12 3. 16 3. 08	2. 0 2. 2 2. 6 2. 5 2. 3	2. 2 2. 4 2. 7 2. 6 2. 3	4. 2 4. 6 5. 3 5. 1 4. 6	46. 3 45. 9 48. 2 48. 5 48. 5	72. 4 72. 9 73. 2 73. 5 73. 7	80. 6 83. 6 83. 0 84. 5 83. 8	84. 0 85. 6 85. 8 84. 5 85. 3	1.5 1.2 1.2 1.4 1.2	85. 5 86. 8 87. 0 85. 9 86. 5			
7 8 10 11 12	3. 06 3. 02 2. 92 2. 89 2. 87	2. 2 2. 5 2. 8 3. 2 3. 0	2.1 2.2 1.9 1.8 1.7	4. 3 4. 7 4. 7 5. 0 4. 7	46.8 47.1 46.9 48.5 49.7	72. 6 72. 4 73. 7 73. 7 74. 0	84. 6 84. 1 84. 5 84. 9 84. 6	85. 6 84. 5 84. 9 85. 0 86. 9	1.1 1.2 1.0 .8 .6	86. 7 86. 7 85. 9 85. 8 87. 5			
Average	3. 03	2. 5	2.3	4.8	47. 7	73. 2	83.8	85. 2	1. 2	86. 4			

<sup>1</sup> Besides the adjusted values for all wave lengths, the observed transmissions and the corrections required in adjusting to a uniform thickness of 2.5 mm (approximately the mean thickness of all the samples for which data are given in Tables 1 and 2) are also included in the cases of the 2 extreme wave lengths employed.

The observed data on which the adjusted and average values are based are all effectively the averages of

The observed data on which the adjusted and average values are based are all effectively the averages of at least 5 determinations which seldom varied over a range as great as 3 per cent in terms of transmission. Consequently, the observed, adjusted, and average values in all these tables should show even smaller spreads. Between the adjusted values for the several samples represented in Tables 1 and 2, the spread (for a given wave length) includes, however, the effect of the somewhat different transmissions of the various samples.

Table 2.—Helio glass

#### INITIAL TRANSMISSIONS 1

		Wave lengths—										
Sample No.	Thick- ness		280		313	334	365		405			
		Ob- served	Correc- tion	Ad- justed	Ad- justed	Ad- justed	Ad- justed	Ob- served	Correc- tion	Ad- justed		
12	mm 2. 27 2. 31	10.9 11.0	2. 2 1. 7	8. 7 9. 3	66. 0 67. 5	81. 2 82. 3	87. 3 89. 4	90. 5 90. 1	0.2	90. 3 89. 9		
3 4 5	2. 30 2. 33 2. 38	11. 3 11. 5 11. 0	1. 9 1. 6 1. 1	9. 4 9. 9 9. 9	63. 2 67. 7 66. 0	82. 6 81. 0 82. 2	89. 1 88. 8 88. 2	89. 5 89. 5 89. 8	.2	89. 3 89. 3 89. 7		
6	2. 39 2. 25 2. 05	10. 3 12. 1 14. 1	1. 0 2. 4 4. 8	9. 3 9. 7 9. 3	65. 5 66. 1 70. 3	81. 8 86. 1 86. 0	88. 9 89. 7 88. 4	90. 3 90. 3 90. 7	.1 .2 .3	90. 2 90. 1 90. 4		
9 10 Average	2. 26 2. 32 2. 29	12. 1 11. 1 11. 5	2. 4 1. 7 2. 0	9. 7 9. 4 9. 5	66. 1 67. 5 66. 5	82. 7 84. 2 82. 9	90. 5 89. 1 88. 9	91. 0 90. 0 90. 2	.2	90. 8 90. 9 90. 0		

<sup>&</sup>lt;sup>1</sup> See note following Table 1.

# Table 2.—Helio glass—Continued TRANSMISSIONS AFTER SOLARIZATION

		Wave lengths—										
Sample No.	mple No. Thickness	280			313	334 365		405				
		Ob- served	Correc- tion	Ad- justed	Ad- justed	Ad- justed	Ad- justed	Ob- served	Correc- tion	Ad- justed		
1 2 3 4 5	mm 2. 27 2. 31 2. 30 2. 33 2. 38	3. 2 3. 7 3. 2 3. 2 2. 8	0.9 .8 .9 .5	2. 3 2. 9 2. 4 2. 3 2. 3	48. 9 49. 3 49. 3 49. 7 50. 0	73. 4 73. 0 72. 9 72. 3 72. 6	82. 7 84. 5 82. 3 84. 0 83. 6	88. 9 88. 5 87. 4 87. 5 88. 4	0. 3 . 3 . 4 . 3 . 2	88. 6 88. 2 87. 0 87. 2 88. 2		
6	2. 39 2. 25 2. 05 2. 26 2. 32	2. 9 3. 4 4. 3 3. 7 3. 6	1. 1 2. 1 1. 1 . 8	2. 5 2. 3 2. 2 2. 6 2. 8	51. 2 50. 8 50. 5 55. 3 56. 4	73. 6 73. 9 72. 4 72. 5 74. 2	84. 7 82. 6 80. 8 84. 3 82. 3	87. 0 88. 7 90. 3 89. 4 89. 8	.2 .4 .4 .3 .2	86. 8 88. 3 89. 9 89. 1 89. 6		
Average	2. 29	3. 4	.9	2. 5	51. 2	73. 1	83. 2	88. 6	.3	88. 3		

Table 3.—Per cent transmission (adjusted to 2.5 mm)
VITA GLASS, LOT B (AVERAGE THICKNESS 2.95 MM)

M-sedment N.	Treat-	Treat-	Wave lengths						
Treatment No.	ment	ment period	280	313	334	365	405		
	Tempera- ture, ° C.	Days							
1	575	1	24. 2	70.0	84.7	89. 1	89. 1		
2	_ 550	1	25. 1	71.0	85. 2	89.8	90.4		
3	_} 530	1	25. 0	70. 5	84.4	88.9	88.8		
4	_ 500	1	24.1	69.9	84.3	89. 2	88.9		
_	477		10.0	00.1	02.0	00 7	88.9		
5	475 450	1	19. 2 19. 4	69. 1 67. 6	83. 2 84. 2	88. 7 88. 9	89. 2		
67		$\frac{1}{2}$	15. 2	65. 4	82. 5	88.5	90.7		
Q	350	6	10.7	61. 4	81.0	87. 7	89. 9		
0	- 350	O .	10.7	01. 1	01.0	01.1	00.0		
9	300	6	7.3	58.6	80.7	88.8	90.1		
10		6	4.9	52. 2	78.7	86. 5	89. 2		
11	_ 600	1	22.1	69.0	84.3	89.4	88.7		
12	_ 200	5	} 20.1	68.3	85. 2	89. 4	89.8		
	515	1	] 20.1	00.0	00. 2	03. 1	00.0		
	1								

## HELIO GLASS, LOT B (AVERAGE THICKNESS 2.25 MM)

			1	1			
1	575	1	12.8	70. 2	84. 3	90.3	90. 5
2	550	1	12.7	70. 2	85. 1	90.8	90.6
3	530	1	13. 7	71. 1	83. 9	90.0	90. 6
4	500	ī	15. 4	69.8	82. 4	89.8	90.0
5	475	1	11.6	69.7	84. 0	89.6	89. 7
6	450	1	13. 1	69. 9	94.6	89.8	89. 9
7	400	2	10.8	67.8	83. 3	88.3	90. 1
8	350	$\bar{6}$	8.9	67.1	81. 7	87. 9	89.7
9	300	6	5.5	64. 2	83. 9	88.6	91.0
10	200	6	4.0	58, 4	78.7	86. 9	89.7
11	600	1	10.8	67. 6	83. 2	90.1	90.1
12	200	5	)		04.1	00.5	00.0
	515	1	12.8	69.3	84.1	89. 5	90.0
			ľ				

Table 4.—Per cent transmission (adjusted to 2.5 mm)
VITA GLASS, LOT A (AVERAGE THICKNESS 3.11 MM)

	1	1								
m - 4 - 1 - 27 -	Treat-	reat- Treat-		Wave lengths						
Treatment No.	ment	ment period	280	313	334	365	405			
1	Tempera- ture, ° C. 200 300 350 400	Days 14 14 13	6. 5 8. 1 9. 6 13. 2	54. 4 57. 8 61. 4 63. 7	77. 7 80. 4 82. 4 83. 9	86. 2 87. 6 89. 2 88. 8	87. 8 88. 4 89. 3 88. 8			
5	450 475 500 525	14 14 14 7	15. 6 17. 9 28. 2 29. 8	63. 9 64. 4 72. 5 70. 3	82. 3 80. 7 84. 1 82. 7	86. 8 86. 7 89. 2 88. 4	87. 86. 89. 88.			
9	550 575 600–610 515	7 7 7 7	25. 3 27. 0 19. 1 22. 2	70. 3 69. 1 62. 2 63. 8	84. 5 84. 7 78. 0 77. 8	88.6 89.3 83.5 83.4	88. 89. 85. 84.			
HELIO GLASS, LOT	A (AVE	RAGE TE	IICKNI	ESS 2.32	MM)					
1	200 300 350 400	14 14 13 14	3. 4 5. 3 6. 6 8. 6	55. 6 62. 1 66. 1 66. 8	76. 3 80. 6 83. 1 82. 3	84. 8 87. 8 89. 6 88. 6	88. : 89. ( 90. :			
5 6	450 475 500 525	14 14 14 7	13. 4 16. 2 15. 6 18. 2	69. 5 70. 0 70. 5 71. 0	81. 8 83. 8 82. 0 84. 2	88. 3 88. 6 89. 4 88. 9	89. 8 90. 3 90. 2 89. 6			
9	550 575 600–610	7 7 7	14. 4 17. 2 10. 1	69. 4 67. 7 62. 9	83. 7 79. 5 78. 2	88. 3 87. 2 87. 9	89. 3 88. 2 86. 4			

13. 8

64. 6

81. 8

86. 9

88. 9

In Figures 3 and 4 the circles indicate the results obtained on lot A for which the succeeding treating temperatures were increased, while the large dots represent the results on lot B, for which the temperatures were decreased. These results give some indication that the treatments from 550° to 600° C. injure the surfaces somewhat, since the transmissions fall off noticeably from the maximum values as the treating temperatures near 600° C. Moreover, the transmissions of lot B for the shortest wave length after the treatments at temperatures from 575° to 500° C. are in general lower than those of lot A, although this discrepancy may, of course, be ascribed to the longer heat treatments received by the latter sets of samples. On the other hand, length of treatment is not always the controlling factor since, from 350° to 475° C., the results for lot A are in many cases lower than those of lot B. This, however, may very well be considered as an indication that the repeated solarization and partial restoration at temperatures below 500° C. caused a condition to develop in the glass that progressively and materially depressed the restoration possible at all temperatures well below this point without greatly affecting the minimum transmissions obtained after the various solarizations. If such a condition does build up as a result of repeated solarization and restoration at low temperatures it is apparently destroyed by heat treatments in the annealing range.

In all other respects (that is, outside of the variations which lead to the possibilities mentioned in the above paragraph) the results are usually as consistent as could be expected in view of the possible experimental errors which include the effect of the impossibility of always being certain that the light traversed exactly the same area of This consistency a sample in the various transmission measurements. indicates that, at the low temperatures especially, the shorter periods of treatment for lot B were sufficient to restore the glass approximately to the maximum possible degree for the various treating temperatures, since otherwise larger differences between the results for the two lots (with those of lot A more generally exceeding those of lot B) would have been observed.

It will be noted that the initial transmissions for the shorter wave lengths are intermediate between those obtained after solarization and those reached after restoration by heat treatments just above That these values after approximately complete restoration at the higher temperatures were above those obtained on the untreated samples is not surprising because the glasses through exposure to light after their production and before being procured for these tests were undoubtedly solarized to some degree. On considering Tables 1 and 2, together with 3 and 4, it also seems that the treatments for reasonably long periods at temperatures very little if any higher than 400° C. were always adequate to restore the solarized glasses to their initial power of transmitting light of the shorter wave lengths, while treatments at temperatures as low even as 200° C. always erased a

noticeable portion of the effect of solarization.

Solarization caused only a relatively small reduction in the transmission at the wave length 405 mµ, and it was consequently difficult to establish any marked difference in the effects due to heat treatment at different temperatures until those temperatures were reached which caused surface deterioration and deformation. It appears, however, that a much greater proportion of the effect of solarization is erased by heat treatments at 200° and 300° C. in the case of the long wave lengths than in that of the short, although the ratio of the maximum effect of either solarization or restoration to the initial transmission usually increases rapidly as the wave length decreases. It appears also that the treating temperature required to restore a solarized glass to its initial transmission for a given wave length decreases as this wave length increases and that this is more apparent in the case of vita glass than in that of helio glass which was found to give the weaker visible thermoluminescence.

#### 2. THERMOLUMINESCENCE

Several tests were made on the thermoluminescence effects which, as is well known, are found after these glasses are solarized, but only those tests made on lots A and B will be mentioned specifically. vita glass samples of lot A, after all the transmission tests had been made and after being again solarized, were heat treated for one week Relatively speaking, the thermoluminescence was very bright and lasted for about 20 minutes, the maximum being reached after about half that time. After this treatment the samples were removed from the furnace so that its temperature might be adjusted to 500° C. for the second part of the test. The samples on being reintroduced at this temperature gave no noticeable glow, which

seemed to indicate that the power to emit visible thermoluminescent radiation had been exhausted by the 300° C. treatment, since solarized glass of this kind when directly introduced into a furnace at 500° C. glows even more brightly than at lower temperatures, although for a shorter time.

The helio-glass samples of lot A when treated according to the same schedule glowed less than the vita glass and for a shorter time at 300° C. They also gave no detectable glow on reheating at 500° C. Since only visual tests were made it is quite possible, however, that both glasses at this temperature emitted radiations of wave lengths

shorter than those of the visible spectrum.

The samples of lot B were tested in much the same manner except that the first treating temperature was 200° C. and the treating period was five days, while the second temperature was 515° C. In this test both glasses were treated together, so that the difference in the brilliance of their glows was more apparent. The more brilliant vita-glass glow reached its maximum after about 20 minutes at 200° C. and lasted at least 3 hours. As before, the period of glowing appeared somewhat shorter in the case of helio glass, but the difference in brilliancy may have been the reason for this. On receiving the added treatment at 515° C. the vita glass glowed very slightly, but this was no longer detectable after 20 minutes, while the helio glass did not seem to glow at all.

The visible radiation obtained in these tests when examined with a small spectroscope appeared to be continuous throughout its spectrum, which seemed not to reach the longest visible wave lengths, while the shorter wave-length regions were relatively bright. As a matter of fact, however, the glows were too weak for definite conclusions on these points unless much more refined methods of observation had been used, and such refinements seemed unnecessary for the purpose

of this investigation.

From these tests it is clearly evident that treatments at relatively low temperatures practically exhaust the power of solarized glasses to glow visibly, while from the transmission measurements, it would seem that they are at the same time relatively more effective in restoring the loss in power of the glasses to transmit radiation at the longer wave lengths than they are in doing the same thing at the shorter Moreover, it was noted from cursory inspections that all the treatments at low temperatures seemed to decolorize the glasses as completely as those at the higher temperatures. This may be an indication that the visible thermoluminescence accompanies the reversal of those processes which during solarization are relatively more effective in decreasing the transmission in the range of the visible and longer ultra-violet wave lengths; and if it should be shown ultimately that an invisible radiation of shorter wave lengths is emitted more intensely by the glass at the higher temperatures it might well be that such radiation accompanies the reversal of other solarization processes which are relatively more potent in decreasing the transmission for the ultra-violet wave lengths near the lower end of the transmission range. In other words, the disappearance of the coloration and the coincident visible thermoluminescence appear to be more closely associated with transmissivity restorations at the longer

ultra-violet wave lengths than they appear to be to similar restorations at the wave lengths included in the therapeutic range.

## V. CONCLUSIONS

In a series of tests made for the purpose of determining the effect of heat treatments at different temperatures on artificially solarized samples of commercial glasses having fairly high transmissivities for ultra-violet radiation, the following conclusions were reached.

1. Heat treatments at temperatures in the annealing range of such glasses are the most effective ones in gaining the highest degree of transmission for ultra-violet radiation; and in the cases of the glasses

tested, such temperatures are somewhat above 500° C.

2. Heating a solarized sample of these ultra-violet transmitting glasses for a few hours at temperatures near or somewhat above 300° C. will often approximately restore the transmissivity to its initial value, since this value is ordinarily considerably less than that which may be reached by treatments at higher temperatures, especially if the glass is one that is easily solarized.

3. Regardless of their length, heat treatments at temperatures below 300° C. are apparently inadequate for restoring the solarized glasses to their original transmission except possibly for relatively

long wave lengths.

4. The visible coloration which appears coincident with solarization of these glasses disappears almost completely with treatments of

reasonable length even at temperatures below 300° C.

5. The power to emit thermoluminescence and the coloration seem to disappear simultaneously; and, after these have disappeared, the transmissivity for wave lengths near the visible spectrum appears to be practically restored although that for those shorter wave lengths which are nearer the limit of the transmission range has by no means undergone a full restoration.

6. As other investigators have shown, the thermoluminescence and coloration disappear more and more rapidly as the temperature is increased; consequently the intensity of the luminescence is greatly increased while the period of its visibility is correspondingly decreased as the treating temperature approaches the annealing range.

7. Likewise, the restoration of the power of transmission is also achieved much more rapidly, as well as more completely, by increasing the temperature of treatment.

Washington, March 1, 1931.