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| <p>(54) Title: NEUTRAL, LOW TRANSMITTANCE GLASS<br/>(57) Abstract<br/>A neutral, generally green-gray low transmittance (no more than 50 percent luminous transmittance) soda-lime-silica glass has reduced solar energy transmittance and reduced ultra violet radiation transmittance when provided with the following colorant constituents in percent by weight: Fe<sub>2</sub>O<sub>3</sub> (total iron) 1.3 - 2, NiO 0.01 - 0.05, CoO 0.02 - 0.04, Se 0.0002 - 0.003, TiO<sub>2</sub> 0.09 - 3. The glass has a ferrous value in the range of 18 to 30.</p>   |           |   |

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TITLE

## NEUTRAL, LOW TRANSMITTANCE GLASS

FIELD OF THE INVENTION

5           The present invention relates to a dark, neutral colored glass that has low visible light transmittance, particularly low ultraviolet radiation transmittance, and low total solar energy transmittance. Although not limited to a particular use, the glass exhibits a  
10 combination of properties that makes it highly desirable for use in privacy glazings such as in the rear portions of vehicles such as vans. These properties include low visible light transmittance to reduce visibility of the contents of the vehicle, low infrared and total solar  
15 energy transmittance to reduce heat gain in the interior of the enclosure, low ultraviolet radiation transmittance to protect fabrics and other interior components from degradation, preferably a neutral green-gray color for the sake of coordinating with a wide range of interior and  
20 exterior finish colors, and a composition compatible with flat glass manufacturing methods, e.g., conventional commercial float glass processes.

BACKGROUND OF THE INVENTION

25           Glasses with good infrared absorption are usually produced by reducing iron present in the glass to the ferrous state or by adding copper. Such materials give glasses a blue color. The materials added to achieve good

ultraviolet radiation absorption are  $\text{Fe}^{3+}$ , Ce, Ti or V. The quantities added to cause the desired level of ultraviolet absorption are such as to tend to color the glass yellow. The combination in the same glass of both good UV and good IR absorption gives glasses whose color is either green or blue. Proposals have been made to produce vehicle glazings with good protection against IR and UV radiation in gray or bronze, but the proposed glasses tend to have a greenish yellow tinge.

Prior art heat absorbing glasses which are neutral and of a blue, green, gray or bronze tint tend to have considerably higher solar light transmittances than would be desirable in privacy glazings. For example, U.S. Reissue Patent No. 25,312 is directed to a gray glass composition containing 0.2 to 1 percent  $\text{Fe}_2\text{O}_3$ , 0.003 to 0.05 percent NiO, 0.003 to 0.02 percent CoO and 0.003 to 0.02 percent Se and having a visible transmittance of between 35 and 45 percent at an 0.25 inch thickness.

A typical dark gray prior art glass has the following composition: 72.9%  $\text{SiO}_2$ , 13.7%  $\text{Na}_2\text{O}$ , .03%  $\text{K}_2\text{O}$ , 8.95% CaO, 3.9% MgO, .10%  $\text{Al}_2\text{O}_3$ , .27%  $\text{SO}_3$ , .06%  $\text{Fe}_2\text{O}_3$ , .015% CoO and .095% NiO. Solar energy absorption of this type of glass is not as low as would be desired for the purposes of the present invention.

More recently nickel-free heat absorbing gray glasses have been proposed for use in low transmittance glazings. Examples of these glasses may be found in U.S. patents Nos. 4,104,076 and 5,023,210. However, both of these

patents include chromic oxide as a coloring agent, may require the use of melting operations/apparatus other than conventional overhead fired tank-type melting furnaces to provide the reducing conditions during melting necessary to produce the desired glasses, and have colorant concentrations of iron oxide, cobalt oxide and selenium that would not produce the particular combination of properties desired here.

10

SUMMARY OF THE INVENTION

We have identified a requirement for excellent neutral privacy glasses such that, in the CIELAB system, they have a color coordinate range:  $a^*$  of  $-5 \pm 5$ , preferably  $-4 \pm 3$ , most desirably  $-4$ ;  $b^*$  of  $10 \pm 20$ , preferably  $0 \pm 10$ , most desirably  $+4 \pm 1$ ; and  $L^*$  of  $50 \pm 10$ , preferably  $50 \pm 5$ , most desirably  $48 \pm 2$ , at visible light transmittances of no more than 50%. More particularly, at a nominal reference thickness of 4mm the glass of the present invention exhibits a visible light transmittance (C.I.E. Illuminant A) of no more than 50%, preferably no more than 25%, and most desirably no more than 20%; a total solar energy transmittance of at least 15 percentage points below the visible light transmittance when the visible light transmittance is within the range of 40 to 50 percent, and a total solar energy transmittance of below 25 percent when the visible light transmittance is below 40 percent, and in any case no more than the visible light transmittance; and an ultraviolet radiation transmittance

of no more than 25%, preferably no more than 18%, and most preferably no more than 15%<sup>1</sup>. These neutral, generally green-gray glasses, having a shading coefficient of no more than .53 and preferably no more than .46, are  
5 produced with a standard soda-lime-silica float glass base composition to which is added iron oxide, cobalt oxide, nickel oxide, titanium dioxide and selenium in certain critical proportions.

As is common in the glass industry, the total iron  
10 contained in a glass composition or batch will be referred to herein as the "total iron expressed as Fe<sub>2</sub>O<sub>3</sub>". When a glass batch is melted, however, some of this amount of total iron is reduced to FeO, while the rest remains Fe<sub>2</sub>O<sub>3</sub>. The balance between ferrous and ferric oxides in the melt  
15 is a result of the oxidation-reduction equilibrium. Reduction of Fe<sub>2</sub>O<sub>3</sub> produces not only FeO, but oxygen gas as well, thus decreasing the combined weight of the two iron compounds in the resultant glass product. Consequently, the combined weight of the actual FeO and Fe<sub>2</sub>O<sub>3</sub> contained  
20 in a resulting glass composition will be less than the batch weight of the total iron expressed as Fe<sub>2</sub>O<sub>3</sub>. For

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<sup>1</sup>Radiation transmittance values disclosed herein are based upon the following wavelength ranges:

|             |          |            |
|-------------|----------|------------|
| Ultraviolet | 300-400  | nanometers |
| Visible     | 380-780  | nanometers |
| Total Solar | 300-2130 | nanometers |

this reason, it shall be understood that "total iron" or "total iron expressed as Fe<sub>2</sub>O<sub>3</sub>", as used herein, means the total weight of iron contained in the glass batch before reduction. It should further be understood that "ferrous value", as used herein, is defined as the weight percent ferrous oxide in the resultant glass divided by the weight percent of total iron expressed as Fe<sub>2</sub>O<sub>3</sub>.

Unless otherwise noted, the term "percent (%)" and "parts", as used herein and in the appended claims, means percent (%) and parts by weight. Wavelength dispersive X-ray fluorescence was used to determine the weight percents of NiO, Co<sub>3</sub>O<sub>4</sub>, TiO<sub>2</sub> and Se and total iron expressed as Fe<sub>2</sub>O<sub>3</sub>. Percent reduction of total iron was determined by first measuring the radiant transmission of a sample at a wavelength of 1060 nanometers, using a spectrophotometer. The 1060 nm transmission value was then used to calculate optical density, using the following formula:

$$\text{Optical density} = \text{Log}_{10} \frac{T_0}{T} \quad (T_0 = 100 \text{ minus estimated loss from reflection} = 92; T = \text{transmission at } 1060 \text{ nm}).$$

The optical density was then used to calculate the percent reduction:

$$\text{percent reduction} = \frac{(110) \times (\text{optical density})}{(\text{Glass thickness in mm}) \times (\text{wt\% total Fe}_2\text{O}_3)}$$

Dark neutral colored glass in accordance with the invention can be produced using iron, cobalt, nickel, titanium and selenium as the colorants in the following ranges:

| Weight Percent of Total Glass               |                 |                 |
|---|-----------------|-----------------|
| Component                                   | Range           | Preferred Range |
| Fe <sub>2</sub> O <sub>3</sub> (total iron) | 1.2 - 2         | 1.3 - 1.6       |
| NiO   | 0.01 - 0.05     | 0.0225 - 0.0285 |
| Co <sub>3</sub> O <sub>4</sub>              | 0.02 - 0.04     | 0.020 - 0.026   |
| TiO <sub>2</sub>                            | 0.09 - 3        | 0.4 - 1         |
| Se  | 0.0002 - 0.0050 | 0.0010 - 0.0020 |
| Ferrous Value                               | 18 - 30%        | 19 - 24%        |

Neutral coloration of a green-gray glass is marked by low excitation purity. The glasses of the invention have an excitation purity less than 15%, and preferably less than 10%. The glasses of the invention yield a relatively narrow range of dominant wavelengths ranging from 490 to 580 nanometers, and preferably from 545 to 570.

#### DETAILED DESCRIPTION

Soda-lime-silica glass may be essentially characterized by the following composition on a weight percentage basis of the total glass:

|                                |          |
|--------------------------------|----------|
| SiO <sub>2</sub>               | 68 - 75% |
| Na <sub>2</sub> O              | 10 - 18  |
| CaO                            | 5 - 15   |
| MgO                            | 0 - 5    |
| Al <sub>2</sub> O <sub>3</sub> | 0 - 5    |
| K <sub>2</sub> O               | 0 - 5    |
| BaO                            | 0 - 5    |

Other minor ingredients, including melting and refining aids such as SO<sub>3</sub>, may also appear in the glass

composition. Small amounts of  $B_2O_3$  have also sometimes been included in flat glass and may be considered optional. To this base glass are added the coloring constituents of the present invention set forth above.

5 In accordance with the present invention, Ilmenite is suitable for use as an ingredient in the glass composition. Ilmenite is optionally used as a titanium source and for supplying at least a partial amount of the  $Fe_2O_3$ .

10 The glasses of the invention are essentially free of colorants other than those specifically recited, with the exception of any trace amounts of such colorants that may be present as impurities. These glasses may be melted and refined continuously in a conventional tank type melting  
15 furnace and formed into flat glass sheets of varying thicknesses by the float method in which the molten glass is supported on a pool of molten metal, usually tin, as it assumes a ribbon shape and is cooled.

Examples 1 through 6 are all satisfactory embodiments  
20 of the invention that exhibit a pleasing neutral green-gray appearance and provide excellent reduction of solar energy transmittance and a very desirable shading coefficient. Shading Coefficient is calculated from Lawrence Berkely Laboratories' Window 3.1 software program  
25 which is based on an outdoor temperature of 89°F (32°C), an indoor temperature of 75°F (24°C), a solar intensity of 248 Btu/(hr x sq ft), (789 W/sq m), and a 7.5 mph (12 kph) wind. Thus, luminous transmittance ("I11 A") is less than

20% for all of these examples at a reference thickness of 4mm. The total solar energy transmittance ("TS") of each example is less than 18% and in all cases is less than the luminous transmittance. The ultraviolet radiation transmittance is exceptionally low in each of the examples, with none exceeding 16.5 percent. The shading coefficient of each example is in all instances not greater than .47.

10

TABLE I

|                                      | Ex. 1 | Ex. 2 | Ex. 3 | Ex. 4 | Ex. 5 | Ex. 6 |
|--------------------------------------|-------|-------|-------|-------|-------|-------|
| Total Fe <sub>2</sub> O <sub>3</sub> | 1.21  | 1.35  | 1.357 | 1.2   | 1.4   | 1.394 |
| % Ferrous                            | 19.7  | 19.7  | 23.4  | 25    | 20    | 20.8  |
| Co <sub>3</sub> O <sub>4</sub> (ppm) | 310   | 206   | 220   | 138   | 230   | 221   |
| % TiO <sub>2</sub>                   | 2.47  | 0.42  | 0.217 | .5    | 1.0   | 0.09  |
| NiO (ppm)                            | 295   | 264   | 266   | 146   | 259   | 268   |
| Se (ppm)                             | 48    | 21    | 15    | 15    | 19    | 15    |
| Ill A                                | 12.7  | 18.4  | 18.9  | 26    | 16.2  | 19.6  |
| TS                                   | 16    | 17.1  | 15.4  | 17    | 10.9  | 17.2  |
| UV                                   | 3.1   | 9.4   | 12.7  | 16.5  | 10.1  | 13    |
| L*                                   | 42.3  | 49.8  | 50.9  | 58.5  | 48.2  | 50.8  |
| a*                                   | -5.8  | -3.39 | -4.36 | -5.1  | -7.7  | -3.8  |
| b*                                   | 9.1   | 6.81  | 1.49  | 0.84  | -3.6  | 1.5   |
| Dom. Wave                            | 566   | 569   | 514   | 502   | 488.9 | 519.4 |
| % Purity                             | 15    | 10.3  | 1.9   | 2.6   | 11    | 1.9   |

Glass made by the float process typically ranges in thickness from about 2 to 10 millimeters. For the preferred solar radiation control features of the present invention, it is preferred that the transmittance properties described herein be obtained within the thickness range of 3 to 5 millimeters.

## WHAT IS CLAIMED IS:

1. A neutral colored soda-lime-silica glass composition comprising on a weight percent basis: 68-75%  
5 SiO<sub>2</sub>, 10-18% Na<sub>2</sub>O, 5-15% CaO, 0-5% MgO, 0-5% Al<sub>2</sub>O<sub>3</sub>, 0-5% K<sub>2</sub>O, and 0-5% BaO and colorants consisting essentially of from about 1.2 to 2 weight percent Fe<sub>2</sub>O<sub>3</sub> (total iron), from about 0.01 to 0.05 weight percent NiO, from about 0.02 to 0.04 weight percent Co<sub>3</sub>O<sub>4</sub>, from about 0.09 to 3 weight  
10 percent TiO<sub>2</sub>, and from 0.0002 to 0.005 weight percent Se, and having a ferrous value in the range of 18 to 30 percent.

2. A glass as defined in claim 1, wherein said  
15 glass, at a nominal 4 millimeter thickness, has a total solar energy transmittance of at least 15 percentage points below the visible light transmittance (Illuminant A) when the visible light transmittance is within the range of 40 to 50 percent, and a total solar energy  
20 transmittance of below 25 percent when said visible light transmittance is below 40 percent.

3. A glass as defined in claim 1, having a color defined by the following CIELAB coordinates: a\*=-5±5;  
25 b\*=10±20; L\*=50±10.

4. A glass as defined in claim 1, having a color defined by the following CIELAB coordinates:  $a^* = -4 \pm 3$ ;  $b^* = +0 \pm 10$ ;  $L^* = 50 \pm 5$ .

5 5. A glass as defined in claim 1, having a dominant wavelength in the range of 490 to 580 and an excitation purity of less than 15.

6. A glass as defined in claim 1, having a dominant  
10 wavelength in the range of 545 to 570 and an excitation purity of less than 10.

7. A glass as defined in claim 1, wherein said  
15 glass has a shading co-efficient no more than 0.53

8. A glass as defined in claim 1, wherein said  
glass has an ultraviolet radiant transmittance of no more  
than 25 percent.

20 9. A neutral colored soda-lime-silica glass  
composition comprising on a weight percent basis: 68-75%  
SiO<sub>2</sub>, 10-18% Na<sub>2</sub>O, 5-15% CaO, 0-5% MgO, 0-5% Al<sub>2</sub>O<sub>3</sub>, 0-5%  
K<sub>2</sub>O, and 0-5% BaO and colorants consisting essentially of  
from about 1.2 to 2 weight percent Fe<sub>2</sub>O<sub>3</sub> (total iron), from  
25 about 0.01 to 0.05 weight percent NiO, from about 0.02 to  
0.04 weight percent Co<sub>3</sub>O<sub>4</sub>, from about 0.09 to 3 weight  
percent TiO<sub>2</sub>, and from 0.0002 to 0.005 weight percent Se,  
and having a ferrous value in the range of 18 to 30

percent; the glass, at a nominal 4 millimeter thickness, having a total solar energy transmittance of at least 15 percentage points below the visible light transmittance (Illuminant A) when the visible light transmittance is  
5 within the range of 40 to 50 percent, and a total solar energy transmittance of below 25 percent when said visible light transmittance is below 40 percent.

10 10. A glass as defined in claim 9, wherein said weight percent  $\text{Fe}_2\text{O}_3$  (total iron) is in the range of 1.3 to 1.6 percent.

15 11. A glass as defined in claim 9, wherein said NiO is present in the range of 0.0225 to 0.0285 weight percent.

12. A glass as defined in claim 9, wherein said  $\text{Co}_3\text{O}_4$  is present in the range of 0.020 to 0.026 weight percent.

20 13. A glass as defined in claim 9, wherein said Se is present in the range of 0.0010 to 0.0020 weight percent.

25 14. A glass defined in claim 9, wherein said  $\text{TiO}_2$  is present in the range of 0.4 to 1 weight percent.

15. A glass as defined in claim 9, having a ferrous value in the range of from about 19 to about 24 percent.

16. A glass as defined in claim 9, having a color defined by the following CIELAB coordinates:  $a^* = -4 \pm 3$ ;  $b^* = 0 \pm 10$ ;  $L^* = 50 \pm 5$ .

5 17. A glass as defined in claim 9, having a shading coefficient of no more than .46.

18. A glass as defined in claim 9, having an ultraviolet radiation transmittance of no more than 18  
10 percent.

19. A neutral colored soda-lime-silica glass composition comprising on a weight percent basis: 68-75% SiO<sub>2</sub>, 10-18% Na<sub>2</sub>O, 5-15% CaO, 0-5% MgO, 0-5% Al<sub>2</sub>O<sub>3</sub>, 0-5% K<sub>2</sub>O, and 0-5% BaO and colorants consisting essentially of  
15 from about 1.3 to 1.6 weight percent Fe<sub>2</sub>O<sub>3</sub> (total iron), from about 0.0225 to 0.0285 weight percent NiO, from about 0.020 to 0.026 weight percent Co<sub>3</sub>O<sub>4</sub>, from about 0.4 to 1 weight percent TiO<sub>2</sub>, and from 0.0010 to 0.0020 weight  
20 percent Se, and having a ferrous value in the range of 18 to 30 percent; the glass having a visible light transmittance of no more than 25 percent, a total solar energy transmittance of no more than 25 percent and an ultraviolet radiation transmittance of no more than 15  
25 percent at a nominal thickness of 4 millimeters.

20. A glass as defined in claim 1, wherein said glass has a shading co-efficient no more than 0.46.

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US96/15133

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|---|--|-----------------------|
| <b>A. CLASSIFICATION OF SUBJECT MATTER</b><br>IPC(6) : CO3C 3/087<br>US CL : 501/70,71<br>According to International Patent Classification (IPC) or to both national classification and IPC   |  |                       |
| <b>B. FIELDS SEARCHED</b><br>Minimum documentation searched (classification system followed by classification symbols)<br>U.S. : 501/70,71<br>Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched<br>Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)   |  |                       |
| <b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>   |  |                       |
| Category*   | Citation of document, with indication, where appropriate, of the relevant passages   | Relevant to claim No. |
| A   | US 5,411,922 A (Jones) 2 May 1995.   |                       |
| A   | US 5,346,867 A (Jones et al.) 13 September 1994.   |                       |
| A   | EP 482,535 A (Longobardo et al.) 29 April 1992.  |                       |
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