

77992/87

APPLICATION ACCEPTED AND AMENDMENTS

AMENDED 16-7-90

COMMONWEALTH of AUSTRALIA

PATENTS ACT 1952

APPLICATION FOR A STANDARD PATENT

602003

I-
We of YAMAMURA GLASS CO., LTD. and KIRIN BEER KABUSHIKI KAISHA
of 2-21, Hamamatsubaracho, 6-26-1, Jingumae,
Nishinomiya-shi, Shibuya-ku,
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JAPAN JAPAN

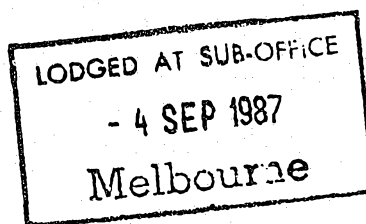
hereby apply for the grant of a Standard Patent for an invention entitled:

"GREEN GLASSES FOR CONTAINERS, CAPABLE OF INTERCEPTING ULTRAVIOLET RAYS AND NEAR ULTRAVIOLET RAYS, AND PROCESSES FOR THE PRODUCTION OF THE SAME"

which is described in the accompanying provisional complete specification.

Details of basic application(s):—

<u>Number</u>	<u>Convention Country</u>	<u>Date</u>
208045/1986	JAPAN	5 September 1986



The address for service is care of DAVIES & COLLISON, Patent Attorneys, of 1 Little Collins Street, Melbourne, in the State of Victoria, Commonwealth of Australia.

Dated this 4th day of September 1987

H. M. Rimington

To: THE COMMISSIONER OF PATENTS

(a member of the firm of DAVIES & COLLISON for and on behalf of the Applicant).

Davies & Collison, Melbourne and Canberra.

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952-1973

DECLARATION IN SUPPORT OF CONVENTION OR
NON-CONVENTION APPLICATION FOR A PATENT
OR PATENT OF ADDITION

Insert title of invention.

In support of the Application made for a ~~patent~~ ^{patent of addition} for an invention
entitled: Green glasses for containers, capable of intercepting
ultraviolet rays and near ultraviolet rays, and processes
for the production of the same

Insert full name(s) and address(es)
of declarant(s) being the appli-
cant(s) or person(s) authorized to
sign on behalf of an applicant
company.

We Takeshi Yamamura and Hideyo Motoyama

of YAMAMURA GLASS CO., LTD. of 2-21, Hamamatsubaracho,
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KIRIN BEER KABUSHIKI KAISHA of 6-26-1, Jingumae,
Shibuya-ku, Tokyo, Japan, respectively

Cross out whichever of paragraphs
1(a) or 1(b) does not apply

1(a) relates to application made
by individual(s)

1(b) relates to application made
by company; insert name of
applicant company.

do solemnly and sincerely declare as follows :-

1. (a) ~~I am~~ the applicant ~~S~~ for the ~~patent~~
We are ~~patent of addition~~

or (b) ~~I am~~ authorized by

YAMAMURA GLASS CO., LTD. and KIRIN BEER KABUSHIKI KAISHA

Cross out whichever of paragraphs
2(a) or 2(b) does not apply

2(a) relates to application made
by inventor(s)

2(b) relates to application made
by company(s) or person(s) who
are not inventor(s); insert full
name(s) and address(es) of inven-
tors.

the applicant ~~S~~ for the ~~patent~~
~~patent of addition~~ to make this declaration on ~~its~~
their behalf.

2. (a) ~~I am~~ the actual inventor ~~S~~ of the invention
~~We are~~

or (b) Yoshihiro ABE of 3-7-59-505, Takakuradai, Suma-ku, Kobe-shi,
Hyogo-ken, Japan;

Masao KITAYAMA of 1-26-22, Nishioike, Kita-ku, Kobe-shi,
Hyogo-ken, Japan;

Naoya MINOURA of Meson Miyama 2-13, 2065-8, Ouzenji,
Asou-ku, Kawasaki-shi, Kanagawa-ken, Japan; and

Akira NOGUCHI of 307, 535, Gosanjo-cho, Hikone-shi,
Shiga-ken, Japan

~~is~~
~~are~~ the actual inventor ~~S~~ of the invention and the facts upon which the applicant ~~S~~
~~is~~
~~are~~ entitled to make the application are as follows :-

The applicants would, if a patent were granted on an application
made by the said inventors, be entitled to have the patent
assigned to them.

State manner in which applicant(s)
derive title from inventor(s)

Cross out paragraphs 3 and 4
for non-convention applications.
For convention applications,
insert basic country(s) followed
by date(s) and basic applicant(s).

3. The basic application..... as defined by Section 141 of the Act ~~was~~ made
in JAPAN on the September 5, 1986
by YAMAMURA GLASS CO., LTD. and KIRIN BEER KABUSHIKI KAISHA
in on the
by
in on the
by

4. The basic application..... referred to in paragraph 3 of this Declaration ~~was~~
the first application..... made in a Convention country in respect of the invention the subject
of the application.

Insert place and date of signature.

Declared at Hyogo-ken, Japan this 14th day of August, 1987
Declared at Tokyo, Japan this 17th day of August, 1987

Signature of declarant(s) (no
attestation required)

YAMAMURA GLASS CO., LTD.

KIRIN BEER KABUSHIKI KAISHA

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Note: Initial all alterations.

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(56) Prior Art Documents
AU 451662 30122/71 19.61
US 4312953
DE 1424911

(57) Claim

1. A green glass for a container containing 0.3 to 1.5% by weight of chromium oxide in terms of Cr_2O_3 , and at most 0.06% by weight of Fe_2O_3 , in a glass composition of SiO_2 - Na_2O - CaO type and having C.I.E. colorimetric values for 10 mm thickness: a brightness (Y) of 1 to 25%, a dominant wavelength (λ_d) of 556 to 564 nm and an excitation purity (Pe) of at least 90%, which is capable of intercepting substantially a light of 340 to 460 nm in a thickness of 4mm; and wherein said glass comprises no other coloring constituents.

COMMONWEALTH OF AUSTRALIA

PATENT ACT 1952

COMPLETE SPECIFICATION

(ORIGINAL)

FOR OFFICE USE

602003

CLASS

INT. CLASS

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Related Art:

This document contains the
amendments made under
Section 49 and is correct for
printing.

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COMPLETE SPECIFICATION FOR THE INVENTION ENTITLED:

"GREEN GLASSES FOR CONTAINERS, CAPABLE OF INTERCEPTING ULTRAVIOLET RAYS AND
NEAR ULTRAVIOLET RAYS, AND PROCESSES FOR THE PRODUCTION OF THE SAME"

The following statement is a full description of this invention,
including the best method of performing it known to us :-

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to green glasses of soda-lime-silica type for containers, capable of absorbing or intercepting ultraviolet rays and near ultraviolet visible rays, and more particularly, it is concerned with green glass compositions for beer bottles, capable of protecting beer from the so-called sunflavor.

2. Description of the Prior Art

It is known that beer is deteriorated by the action of sunlight to give out the sunflavor. In particular, a light within a wavelength range of 400 to 460 nm reacts with bitter substances of hops so that the beer loses the intrinsic amber color and becomes red-tinted, thus giving out a skunky odor, namely sunflavor.

A beer bottle formed of the ordinary amber glass (thickness: about 4 mm, shown in Fig. 1 and Fig. 2, Curve C) intercepts the light of 400 to 460 nm, but another beer bottle of the ordinary green glass does not have properties comparable to those of the amber glass, as shown in Fig. 1, although various efforts have been made to intercept ultraviolet rays. Therefore, beer in a bottle of the green glass must be stored with greater care than in the case of the amber glass. If not so, there arises a problem that the beer early deteriorates.

5 An example of the ordinary green glass commonly used for beer bottles is a glass having a composition comprising 72.2 % SiO_2 , 12.7 % Na_2O , 10.8 % CaO , 1.94 % Al_2O_3 , 1.34 % K_2O , 0.13 % Cr_2O_3 , 0.15 % Fe_2O_3 , % being by weight, and unavoidable impurities, and having a brightness (Y) of 33.8 %, a dominant wavelength (λ_d) of 558.7 nm and an excitation purity (Pe) of 68.3 %. This ordinary green glass intercepts a light of 400 nm, but transmits considerably a light of 400 to 460 nm, as is evident from the percent transmission shown in Fig. 1, Curve D.

10 In the production of such an ultraviolet light intercepting green glass, furthermore, there are various problems due to necessity of maintaining hexavalent chromium in the glass. That is, since an oxidant such as sodium nitrate is used in a proportion of more than 1 part by weight to 100 parts by weight of silica sand in the glass composition, (1) seeds or small bubbles tend to occur in the glass, (2) the production cost is increased because of raising the temperature of a melting furnace or decreasing the production quantity of a melting furnace so as to control the occurrence of seeds and (3) nitrogen oxides (NO_x) are formed through decomposition of the sodium nitrate. Since the operation is carried out by feeding a larger amount of air to secure a more oxidizing state than in the case of the ordinary transparent or colored glass, (1) the energy consumption is consequently increased and the production cost

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is increased and (2) the quantity of nitrogen oxides is increased, resulting in a problem of environmental pollution.

SUMMARY OF THE INVENTION

5 It is an object of the present invention to provide an improved green glass of soda-lime-silica type, for a container, which gives an adequate light protection.

10 It is another object of the present invention to provide a green glass composition capable of intercepting or absorbing ultraviolet rays and near violet visible rays and suitable for the production of containers used for packaging food or beverage.

15 It is a further object of the present invention to provide a beer bottle made from a green glass free from seeds or small bubbles and capable of protecting beer from sunflavor.

20 It is a still further object of the present invention to provide a process for the production of an improved green glass of soda-lime-silica type, whereby the disadvantages of the prior art can be overcome.

25 These objects can be attained by a green glass for a container containing 0.3 to 1.5 % by weight of chromium oxide in terms of Cr_2O_3 and at most 0.06 % by weight of Fe_2O_3 in a glass composition of SiO_2 - Na_2O - CaO type and having C.I.E. colorimetric values for 10 mm thickness: a brightness (Y) of 1 to 25 %, a dominant wavelength (λ_d) of 556 to 564

nm and an excitation purity (Pe) of at least 90 %, which is capable of intercepting substantially a light of 340 to 460 nm in a thickness of 4 mm.

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BRIEF DESCRIPTION OF THE DRAWINGS

The accompanying drawings are to illustrate the principle and merits of the present invention in greater detail.

Fig. 1 and Fig. 2 are respectively graphs showing the percent transmissions of lights in the ultraviolet region and near ultraviolet visible region as to green glasses of Example 1 to Example 3 according to the present invention, and amber glass and green glass of the prior art, each having a thickness of 4 mm, except Curve F (1 mm) (Fig. 1) and 2 mm (Fig. 2).

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DETAILED DESCRIPTION OF THE INVENTION

The inventors have made various efforts to develop a green glass for a container capable of preventing photochemical deterioration of the content, in particular, protecting beer from the so-called sunflavor and consequently, have succeeded in obtaining such a green glass by the use of a silica sand for transparent glasses, which has a smaller Fe_2O_3 content.

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Accordingly, the present invention provides a green glass for a container containing 0.3 to 1.5 % by weight,

preferably 0.35 to 1.5 % by weight, in particular, 0.35 to 0.5 % by weight of chromium oxide in terms of Cr_2O_3 and at most 0.06 % by weight of Fe_2O_3 in a glass composition of SiO_2 - Na_2O - CaO type, having C.I.E. colorimetric values for 10 mm thickness: a brightness (Y) of 1 to 25 %, preferably 10 to 25 %, a dominant wavelength (λ_d) of 556 to 564 nm and an excitation purity (Pe) of at least 90 % and being capable of substantially intercepting a light of 340 to 460 nm in a thickness of 4 mm, and a process for the production of the above described green glass, which process comprises preparing and melting a batch composition comprising 100 parts by weight of silica sand, 24 to 40 parts by weight of soda ash, 20 to 30 parts by weight of limestone, 0.5 to 3.0 parts by weight of sodium sulfate, 0.8 to 4.0 parts by weight of a bichromate, 0 to 3 parts by weight, preferably 0 to 1 part by weight of a nitrate and unavoidable impurities.

Generally, the glass composition of SiO_2 - Na_2O - CaO type used in the green glass of the present invention comprises 60 to 75 % by weight of SiO_2 , 10 to 16 % by weight of Na_2O , 8 to 12 % by weight of CaO , 0.5 to 6 % by weight of Al_2O_3 , 0 to 5 % by weight of K_2O and unavoidable impurities.

In the green glass composition of the present invention, chromium oxide is added in a proportion of 0.3 to 1.5 % by weight, preferably 0.35 to 1.5 % by weight in terms of Cr_2O_3 , for example, by the use of a bichromate such as potassium bichromate, and the iron content is adjusted

to 0.06 % or less as Fe_2O_3 . The thus added chromium is preferably retained in the hexavalent chromium state as more as possible. The iron components unavoidably come from the silica sand and limestone and are added to the glass composition. When using the ordinary silica sand and limestone for colored glasses, the content of Fe_2O_3 is about 0.15 to 0.2 % by weight, whereas in the present invention, a silica sand for transparent glasses, containing a smaller amount of Fe_2O_3 is particularly used to adjust the iron content to described above.

In the green glass of the present invention, no other coloring constituents than chromium oxides, for example, metal oxides such as CuO , Co_3O_4 , NiO , As_2O_5 , Fe_2O_3 , MnO , CeO_2 and Sb_2O_3 are added. When these metal oxides are added, transmission of heat rays is lowered, resulting in deterioration of the melting and forming property, and Cr^{6+} is converted into Cr^{3+} so that interception of a light near 450 nm is insufficient.

The amount of an oxidant such as sodium nitrate is generally 0, but if necessary, the oxidant can be added in a proportion of less than 1 part by weight to 100 parts by weight of silica sand. Even if the operation is carried out under the ordinary conditions except using the oxidant in such a smaller proportion including 0, a glass capable of substantially intercepting a light of 340 to 460 nm can be obtained.

In the glass composition of the present invention, chromium oxides are contained in a proportion of 0.3 to 1.5 % by weight, preferably 0.35 to 1.5 % by weight, because the green color is more thickened to intercept a light of 400 to 460 nm by increasing the content thereof to more than that of the prior art green glass. If less than 0.3 % by weight, in particular, 0.35 % by weight, the interception of a light of 400 to 460 nm is insufficient, as shown by the percent transmission curve of Comparative Example 1 in Fig. 1, while if more than 1.5 % by weight, the brightness of the glass is excessively lowered. When 1.5 % by weight of chromium oxides are contained in the glass composition, a light protection effect can be given even in the case of a thickness of 1 mm, which effect is substantially similar to the case of adding 0.35 % by weight of Cr_2O_3 for a thickness of 4 mm.

If the amount of iron oxides exceeds 0.06 % by weight as Fe_2O_3 , Cr^{6+} is converted into Cr^{3+} depending on the increment thereof and the interception of a light near 450 nm will probably be insufficient. Since it is assumed that the hexavalent chromium has a large influence upon the interception of a light of near 450 nm, as described above, Cr^{6+} is fed in the form of a bichromate and the content of Fe_2O_3 is controlled to be decreased and metal oxides such as CuO are not added to secure the hexavalent chromium state. Furthermore, the melting and forming property of the glass is deteriorated

depending on the increment of Fe_2O_3 . On the other hand, the amount of an oxidant can largely be decreased by decreasing the content of Fe_2O_3 to a greater extent as compared with the colored glass of the prior art, namely from 0.15-0.2 % by weight to less than 0.06 % by weight, thereby obtaining a melt excellent in fining and a seed-free glass.

According to the present invention, the following advantages can be given:

(1) The content of Cr_2O_3 is increased to thicken green color thereby completely intercepting near ultraviolet visible rays of near 400 to 460 nm and preventing occurrence of the so-called sunflavor.

(2) The amount of an oxidant can be decreased, resulting in a melt excellent in fining, and a green glass free from seeds can thus be produced by largely decreasing the content of Fe_2O_3 .

(3) Production of a green glass can be carried out without lowering the percent transmission of heat rays, deteriorating the melting and molding property of the glass and lowering the production speed by decreasing the content of Fe_2O_3 and excluding addition of metal oxides such as copper oxide.

(4) In the case of a thickness of 2 mm, the percent transmission of the glass according to the present invention in the wavelength region of ultraviolet to 460 nm

is smaller than that of amber glasses of the prior art. Therefore, the glass of the present invention can widely be applied to containers or bottles capable of protecting the contents such as food and beverage.

5 (5) Under the situation requiring light weight containers, the glass composition of the present invention will more favorably be applied. In the case of a thickness of 1 mm, for example, the present invention can display capacity by increasing chromium oxides up to 1.5 % by weight in terms of Cr_2O_3 .

10 (6) Since the use of excessive air and sodium nitrate is controlled according to the present invention and the quantity of NO_x generated in the process can be largely decreased, the problem of the environmental pollution can be solved.

15 The following examples are given in order to illustrate the present invention in detail without limiting the same, in which parts and percents are to be taken as those by weight unless otherwise indicated.

20 Example 1

To 100 parts of silica sand containing 0.04 % of Fe_2O_3 were added 28.8 parts of soda ash, 24 parts of limestone containing 0.025 % of Fe_2O_3 , 1.0 part of sodium sulfate and 1.2 parts of potassium bichromate and the resulting mixture was melted to prepare a green glass having a com-

position of 72.1 % SiO_2 , 13.6 % Na_2O , 10.2 % CaO , 1.8 % Al_2O_3 , 1.5 % K_2O , 0.46 % Cr_2O_3 , 0.04 % Fe_2O_3 and trace unavoidable impurities. This glass had C.I.E. colorimetric values consisting of the following for a typical 10 mm specimen: a
5 brightness (Y) of 16.6 %, dominant wavelength (λ_d) of 560.4 nm and excitation purity (Pe) of 97.8 % and had a percent photo-transmission as shown in Fig. 1 in the case of a thickness of 4 mm (Curve A). It will clearly be understood from the results that the above described green glass is more suitable for a beer bottle since the percent transmission in the case of a thickness of 4 mm corresponding to the thickness of the beer bottle is zero for a light of 400 to 460 nm strongly related with the sunflavor of beer, in comparison with other comparative glasses, Curves D and E in Fig. 1.

15 A glass bottle with a volume of 350 ml, weight of 240 g and side wall thickness of 2.5 mm was made from the above described green glass composition and an emerald green bottle with the same dimension, corresponding to an ultra-violet ray intercepting green glass as shown in Fig. 1, Curve
20 D, was used for comparison. Both the bottles were filled with beer and then subjected to a sensuous test in which periods of time were measured until their sunflavors reached a same level. The green glass bottle of the present invention showed a period of time of 3.9 times as long as the
25 comparative bottle.

Example 2

The mixing and melting procedure of Example 1 was repeated except decreasing the quantity of the potassium bichromate to 0.9 part thus obtaining a green glass with a composition comprising 72.1 % SiO_2 , 13.6 % Na_2O , 10.2 % CaO , 1.8 % Al_2O_3 , 1.5 % K_2O , 0.35 % Cr_2O_3 , 0.06 % Fe_2O_3 and trace unavoidable impurities. The thus resulting glass had C.I.E. colorimetric values consisting of the following for a typical 10 mm specimen: a brightness (Y) of 23.9 %, a dominant wavelength (λ_d) of 561.7 nm, and an excitation purity (Pe) of 96.2 % and had a percent photo-transmission as shown in Fig. 1 in the case of a thickness of 4 mm (Curve B). Similarly, this green glass is suitable for a beer bottle.

The green glasses of Examples 1 and 2 each having a thickness of 2 mm showed percent photo-transmissions shown in Fig. 2, Curves A and B, which were favorably compared with an amber glass, Curve C and the emerald green glass, Curve D, for a light of 400 to 460 nm.

Comparative Example 1

The procedure of Example 1 was repeated except using 0.6 part of potassium bichromate and additionally using 3.0 parts of sodium nitrate, thus obtaining a glass having a composition shown in Table 1. The percent transmission of the glass of Comparative Example 1 is shown in Fig. 1, Curve E.

Example 3

The procedure of Example 1 was repeated except increasing the quantity of the potassium bichromate in the raw materials used in Example 1 to 3.9 parts, thus obtaining a glass with a composition of 72.1 % SiO_2 , 13.6 % Na_2O , 10.2 % CaO , 1.8 % Al_2O_3 , 1.5 % K_2O , 1.5 % Cr_2O_3 , 0.04 % Fe_2O_3 and trace unavoidable impurities. This glass had C.I.E. colorimetric values consisting of the following for a 1 mm specimen: a brightness (Y) of 49.1 %, dominant wavelength (λ_d) of 565.0 nm and excitation purity (Pe) of 88.0 %, and had a percent transmission at a thickness of 1 mm, as shown in Fig. 1, Curve F.

Table 1

(parts by weight)

		<u>Example 1</u>	<u>Example 2</u>	<u>Example 3</u>	<u>Comparative Example 1</u>
	<u>Raw Material Composition</u> ¹⁾				
5	silica sand	100	100	100	100
	soda ash	28.8	28.8	28.8	28.8
	limestone	24.0	24.0	24.0	24.0
	sodium sulfate	1.0	1.0	1.0	1.0
	potassium bichromate	1.2	0.9	3.9	0.6
10	sodium nitrate	0	0	0	3.0
	<u>Glass Composition</u>				
	Cr ₂ O ₃	0.46	0.35	1.5	0.23
	Fe ₂ O ₃	0.04	0.06	0.04	0.04
15	<u>Furnace Condition</u>	normal ²⁾	normal	normal	normal

Note: 1) silica sand for transparent glass

2) "Normal" indicates that the ratio of a fuel and combustion air used in a melting furnace (air-fuel ratio) is in the optimum condition in which the O₂ concentration in the exhaust gas is 0.5 to 3 %.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A green glass for a container containing 0.3 to 1.5% by weight of chromium oxide in terms of Cr_2O_3 and at most 0.06% by weight of Fe_2O_3 in a glass composition of SiO_2 - Na_2O - CaO type and having C.I.E. colorimetric values for 10 mm thickness: a brightness (Y) of 1 to 25%, a dominant wavelength (λ_d) of 556 to 564 nm and an excitation purity (Pe) of at least 90%, which is capable of intercepting substantially a light of 340 to 460 nm in a thickness of 4mm; and wherein said glass comprises no other coloring constituents.

2. A process for the production of the green glass, as claimed in Claim 1, which process comprises preparing and melting a batch composition comprising 100 parts by weight of silica sand, 24 to 40 parts by weight of soda ash, 20 to 30 parts by weight of limestone, 0.5 to 3 parts by weight of sodium sulfate, 0.8 to 4 parts by weight of a bichromate, 0.3 parts by weight of a nitrate and unavoidable impurities.

3. The process as claimed in Claim 2, wherein the silica sand is one for transparent glasses, containing at most 0.06% by weight of Fe_2O_3 .

4. The process as claimed in Claim 2, wherein the batch composition does not contain metal oxides other than chromium oxides.

5. The process as claimed in Claim 4, wherein the metal oxides are CuO , Co_3O_4 , NiO , As_2O_3 , Fe_2O_3 , MnO , CeO_2



and Sb_2O_3 .

6. A green glass as claimed in Claim 1, substantially as hereinbefore described with reference to the Examples and/or drawings.

7. A process for the production of green glass as claimed in any one of Claims 1 to 5, substantially as hereinbefore described with reference to the Examples and/or drawings.

DATED this 10th day of July, 1990

KIRIN BEER KABUSHIKI KAISHA and
YAMAMURA GLASS CO., LTD.

by their Patent Attorneys

DAVIES & COLLISON



FIG. 1

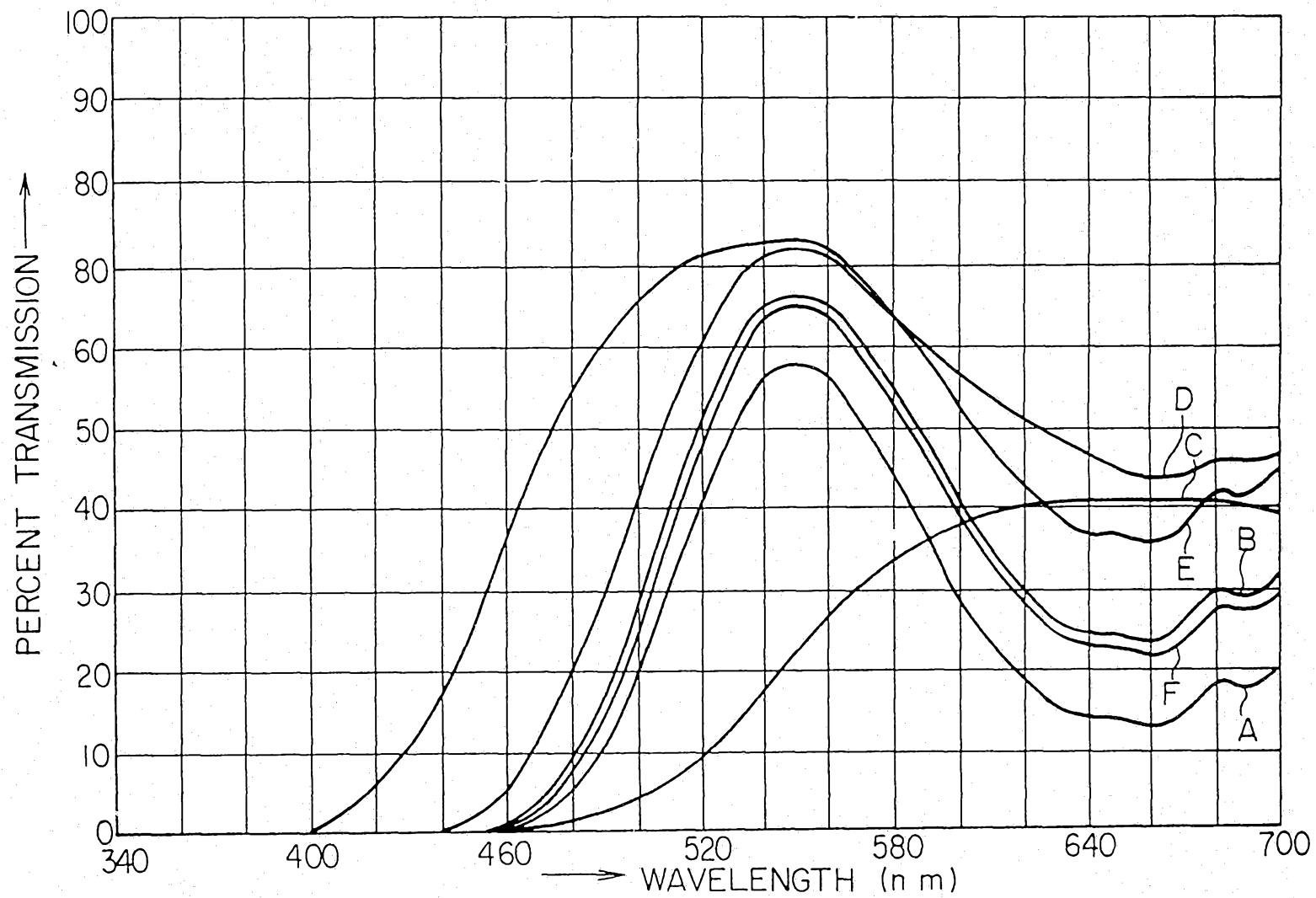


FIG. 2

