

Gamma Ray Interaction with Some High-Lead Glasses Containing Chromium Ions

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Optical absorption of some selected high-lead borate glasses containing chromium exposed to different doses of gamma-irradiation was studied. The results obtained showed that the absorption bands before and after irradiation exhibited changes with the radiation dose and chemical composition of the glass. The response of the glass to gamma irradiation was related to the creation and spread of defects “color centers,” the approach of a saturation condition after a certain irradiation dose, and the possible photochemical effect on the transition metal present in the glass. © 2002 Elsevier Science (USA)

1. INTRODUCTION

The interaction of X-ray or gamma-radiations with glass results mainly in the release of electrons through the Compton process. The resulting electrons or hole traps may be preferentially trapped in structural defects such as vacancies in the interstitial atoms and the multivalent impurities or the nonbridging oxygens. These electrons or hole traps will thus have a new electronic configuration which can cause preferential light absorption and are thus called color centers (1,2). The formation and the nature of the color centers in many different types of glasses have received considerable attention and have been studied extensively by many workers (2–5). The induced colors after exposure to radiation probably resemble the coloring of the ionic crystals, although the random structure of the glass means that there are more possibilities for forming color centers. Thus, the radiation-induced absorption in glass generally consists of overlapping absorption bands rather than discrete bands as are formed in the ionic crystals. During the irradiation of the glass, many polyvalent elements, e.g., Fe and Mn, trap charges either to form optical absorption bands or to inhibit the formation of those bands that occur in the nominally pure glasses (3). Since some of these elements are common impurities in the glass, the reducing or oxidizing conditions of the glass affect their oxidation states, which, in turn, affect the concentration of the various defect centers formed during irradiation (4). Recently, there have been a number of

studies on the effect of the ionizing radiations on the optical properties of glasses containing one of the traditional transition metal ions such as cobalt, nickel, titanium, and copper (5–8).

With respect to chromium-containing glasses, few recent studies were published. Ghoneim *et al.* (9) studied the radiation-induced visible centers in some cabal glasses containing chromium and stated that the optical absorption of the glasses studied shows an increase in the intensity of the absorption band due to Cr^{3+} ions at 600–650 nm with the increase in the irradiation dose. In continuation, it was stated that (10) on increasing the irradiation dose in some selected silicate glasses containing chromium the intensity of the absorption bands gradually increased with progressive marked shifts toward longer wavelengths; the observed induced bands were considered to be associated with hole-trapped defects. El-Batal and El-Din (11) studied the interaction of gamma rays with some borate glasses containing chromium and concluded that the visible absorption bands before and after gamma-ray radiation exhibited changes with the irradiation dose and/or the chemical composition of the glass.

This recent paper deals with the study of gamma-ray-induced optical absorption in some selected lead glasses containing chromium. Special emphasis is placed on comparing the changes in the intrinsic and induced optical absorption in relation to the changes in the glass structure and exposure dose.

2. EXPERIMENTAL

2.1. Glass Samples Preparation

The glasses were prepared from chemically pure materials in amounts sufficient to produce 100 g of glass. Boric oxide was introduced as orthoboric acid. Lead oxide was introduced in the form of red lead oxide. Lithia, soda, potash, magnesia, lime, strontium, and barium oxides were introduced in the form of their respective anhydrous carbonates while zinc oxide, alumina, and chromium oxide were introduced as such.

TABLE 1
Glass Composition of a Number of Lead Borate Glasses

Glass No.	Glass composition (wt %)				
	PbO	B ₂ O ₃	R ₂ O	RO	R ₂ O ₃
B1	70	30			
B2	80	20			
B3	90	10			
B4	75	20	Li ₂ O ≡ 5%PbO		
B5	75	20	Na ₂ O ≡ 5%PbO		
B6	75	20	K ₂ O ≡ 5%PbO		
B7	75	20	MgO ≡ 5%PbO		
B8	75	20	ZnO ≡ 5%PbO		
B9	75	20	CaO ≡ 5%PbO		
B10	75	20	SrO ≡ 5%PbO		
B11	75	20	BaO ≡ 5%PbO		
B12	80	18			Al ₂ O ₃ ≡ 2%B ₂ O ₃
B13	80	15			≡ 5%B ₂ O ₃

Note. All replacement were made on the cation for cation basis.

Batches were melted in Pt-2%Rh crucibles in an electrically heated furnace over a temperature range of 1110 to 1300°C, according to the glass composition, for 3 h. Each melt was rotated several times to produce satisfactory

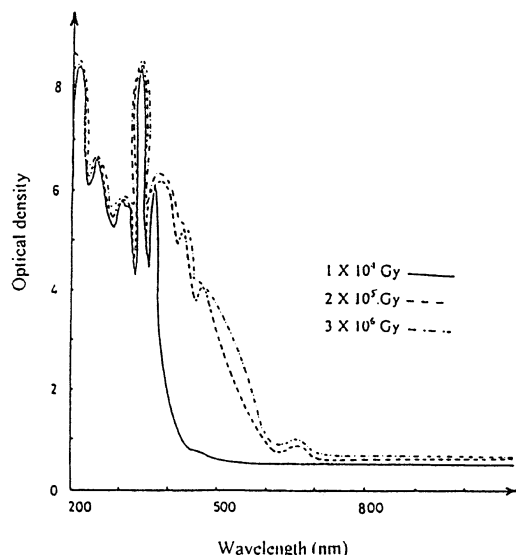


FIG. 1. Absorption spectra of the parent lead borate glass without chromium.

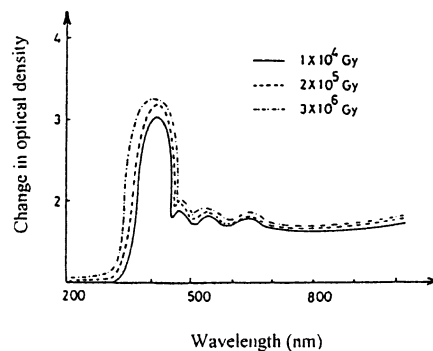


FIG. 2. Induced spectra of the parent lead borate glass.

homogeneity. After complete melting, the melt was cast as rectangular slabs ($\sim 1 \times 1 \times 4$ cm) and properly annealed at the appropriate temperature (400–450°C) in a muffle furnace. Grinding and polishing were carried out in the usual way but with a minimum amount of water and in the final stages of polishing, paraffin oil was used.

2.2. Optical Absorption Measurements

Optical absorption before and 15 min after gamma irradiation was measured using a Shimadzu (Japan) Type 1

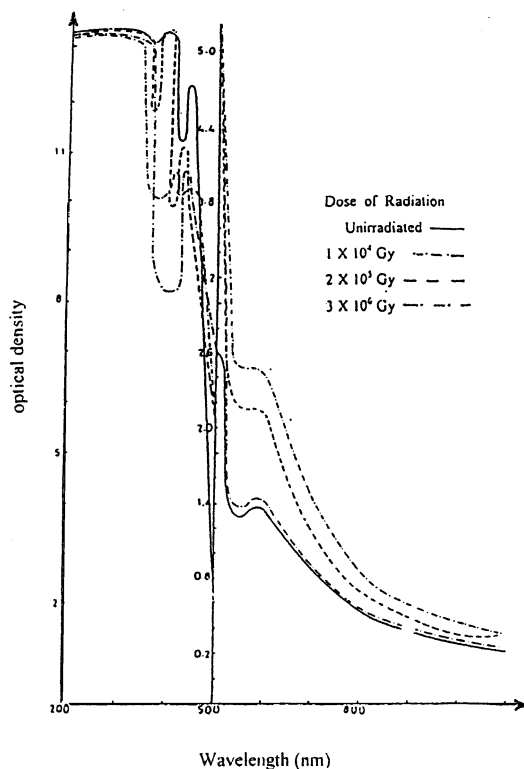


FIG. 3. Absorption spectra of the gamma-irradiated binary lead borate glass of the composition PbO 70%, B₂O₃ 30%, containing 0.05% Cr₂O₃.

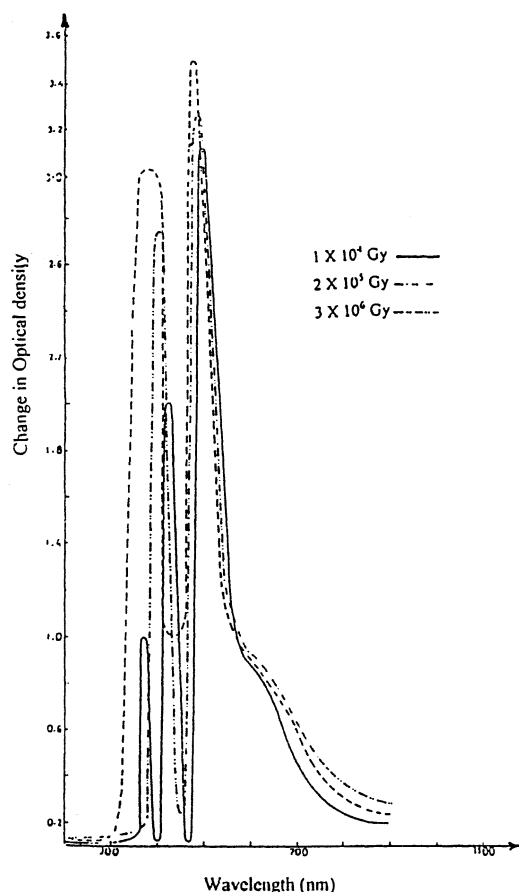


FIG. 4. Induced spectra of the binary lead borate glass of the composition PbO 70%, B₂O₃ 30%, containing 0.05% Cr₂O₃.

spectrophotometer over a wavelength range of 200 to 1100 nm.

2.3. Irradiation Procedure

An Indian ⁶⁰Co source of the gamma cell was used for irradiation. It had a dose rate of 1–5 Gy/s.

3. RESULTS

Spectrophotometric studies were carried out on a number of lead borate glasses containing chromium oxide before and after subjecting the glasses to various progressive doses of gamma rays. The effect of the following factors was evaluated:

1. The composition of the base glass.
2. The radiation dose.

3.1. Lead Oxide–Boric Oxide Glasses

The compositions of the studied binary PbO–B₂O₃ glasses are given in Table 1. The absorption spectra of these

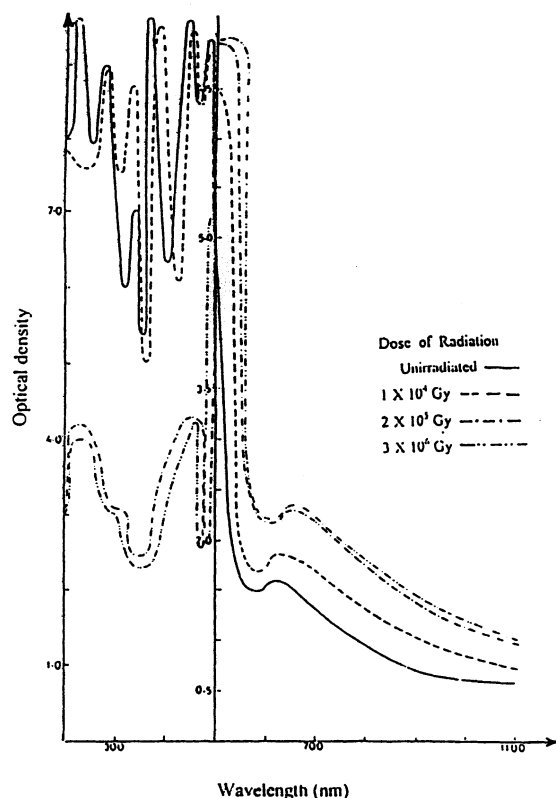


FIG. 5. Absorption spectra of the gamma-irradiated binary lead borate glass of the composition PbO 80%, B₂O₃ 20%, containing 0.05% Cr₂O₃.

glasses before and after gamma irradiation are shown in Figs. 1–8, from which the following can be seen.

a. The parent binary PbO–B₂O₃ glasses without chromium oxide were almost colorless before irradiation; no

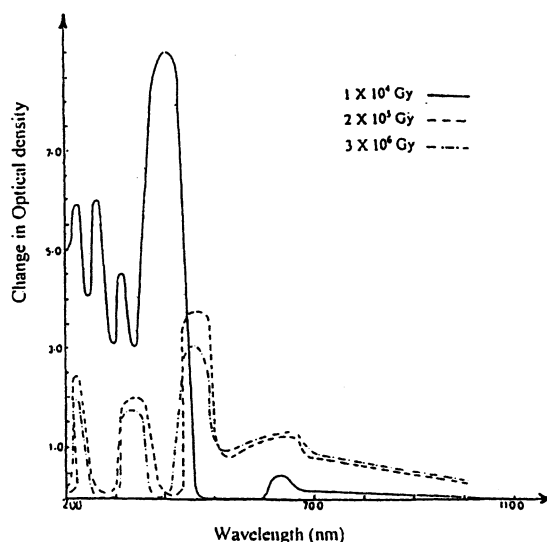


FIG. 6. Induced spectra of the binary lead borate glass of the composition PbO 80%, B₂O₃ 20%, containing 0.05% Cr₂O₃.

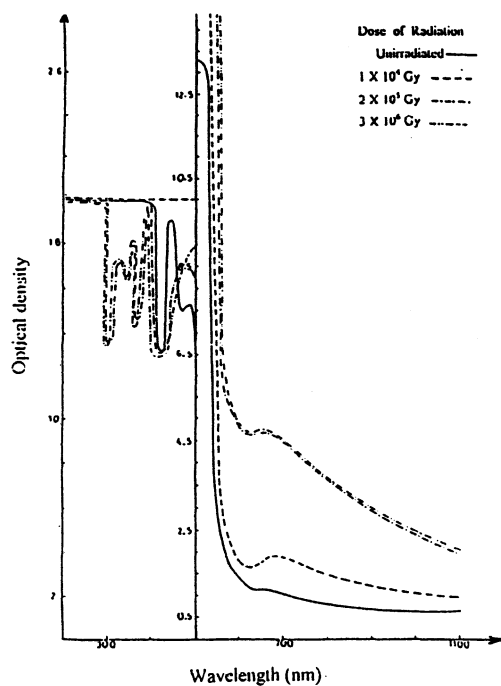


FIG. 7. Absorption spectra of the gamma-irradiated binary lead borate glass of the composition PbO 90%, B₂O₃ 10%, containing 0.05% Cr₂O₃.

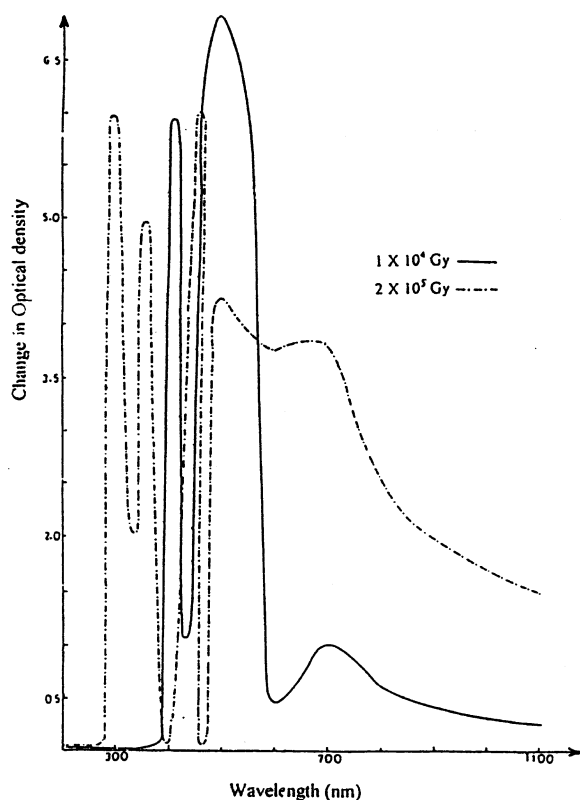


FIG. 8. Induced spectra of the binary lead borate glass of the composition PbO 90%, B₂O₃ 10%, containing 0.05% Cr₂O₃.

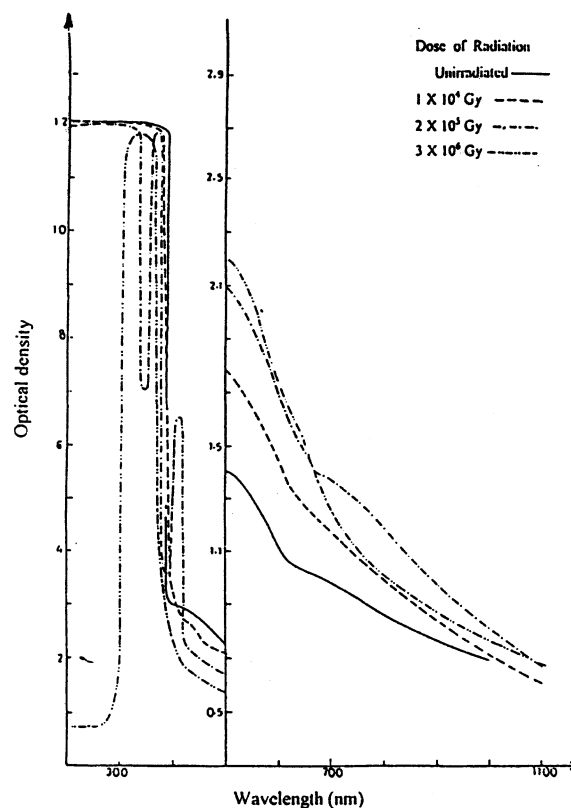


FIG. 9. Absorption spectra of the gamma-irradiated ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, Li₂ 5%, containing 0.05% Cr₂O₃.

characteristic visible absorption bands could be seen in their absorption spectra but some ultraviolet peaks could be observed (Fig. 1). On exposure to gamma rays, the glasses darkened and several absorption bands at about 420, 480, 540, and 650 nm were observed (Fig. 2).

b. In the lead borate glasses with varying chemical compositions ranging from PbO 70% up to PbO 90% (wt%) containing 0.05 g Cr₂O₃/100 g glass, the absorption spectra extending from the ultraviolet to the visible region of the spectrum have been obtained (Figs. 3, 5, and 7); the spectra consist of several absorption bands which were observed in all the glasses studied. The main features of the spectra reveal two visible absorption bands at ≈ 420 and ≈ 620 nm. An extended ultraviolet cutoff was also observed which had been mostly identified to consist of three absorption bands at about 240, 290, and 360 nm.

c. The induced absorption spectra represented in Figs. 4, 6, and 8 showed several induced absorption bands at about 240, 290, 330, 370, 480, and 650 nm which varied in their positions and intensities with both the lead oxide content and the irradiation dose. Most of the experimentally induced absorption bands were repeatable to that obtained from the parent binary lead borate glasses without chromium oxide. The visible absorption band at about 650 nm

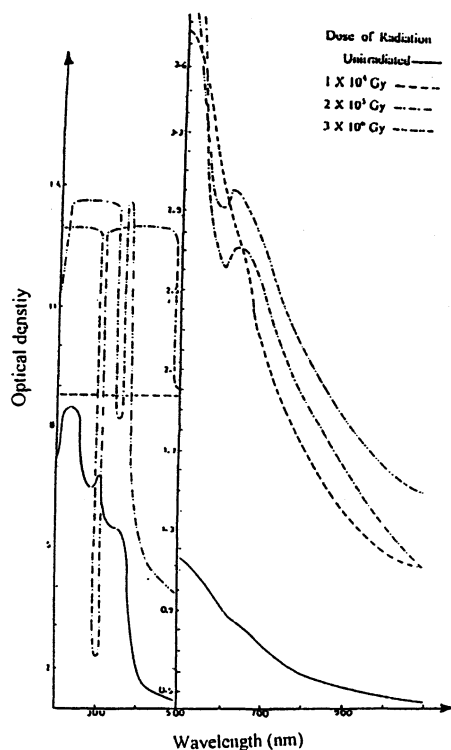


FIG. 10. Absorption spectra of the gamma-irradiated ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, Na₂O 5%, containing 0.05% Cr₂O₃.

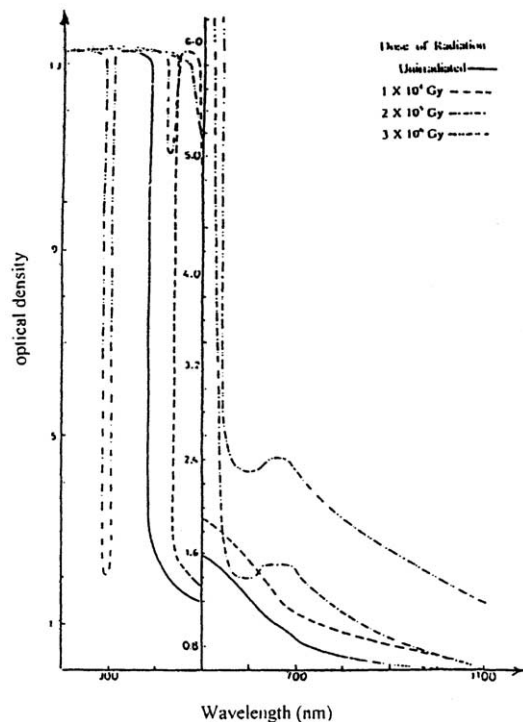


FIG. 11. Absorption spectra of the gamma-irradiated ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, K₂O 5%, containing 0.05% Cr₂O₃.

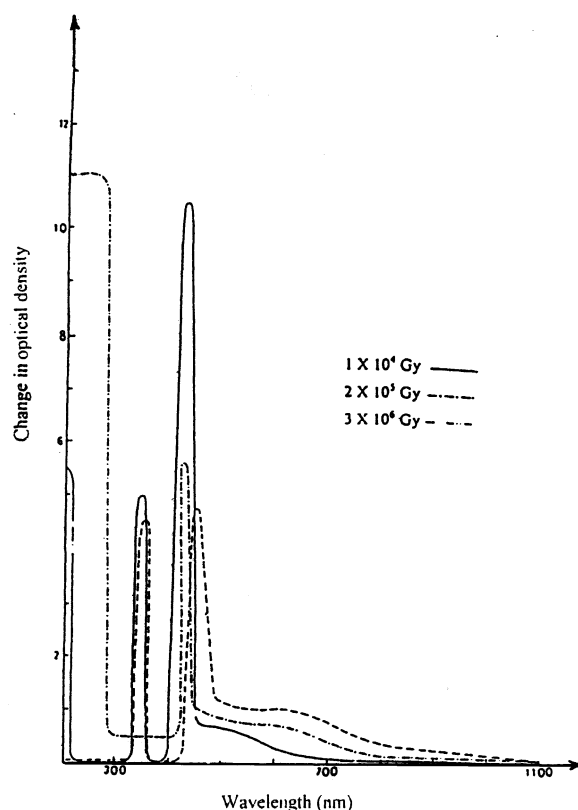


FIG. 12. Induced spectra of the ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, Li₂O 5%, containing 0.05% Cr₂O₃.

shifts to 700 nm in the glass containing the highest lead oxide content (Fig. 8).

3.2. Effect of the Monovalent Metal Oxides

The results obtained from the glass of the base composition (PbO, 80%; B₂O₃, 20% (wt%)) containing 0.05, Cr₂O₃/100 g glass, in which five parts of lead oxide were replaced by one of the monovalent metal oxides, lithia, soda, or potash, cation for cation (Table 1), are shown in Figs. 9–11, from which the following can be seen.

- The absorption band at 600–650 nm was hardly identified in the glass containing soda or potash, while in the glass containing lithia, it was clearly observed.
- The absorption band at ≈ 420 nm almost disappeared in all the glasses containing alkali oxides.
- The ultraviolet absorption band at 300–350 nm was not changed.
- On exposure of the glasses studied to gamma-ray radiation, it can be seen that:

- all the visible absorption bands were easily identified; and
- the intensity of the observed absorption bands increased with the progressive increase of the gamma irradiation dose.

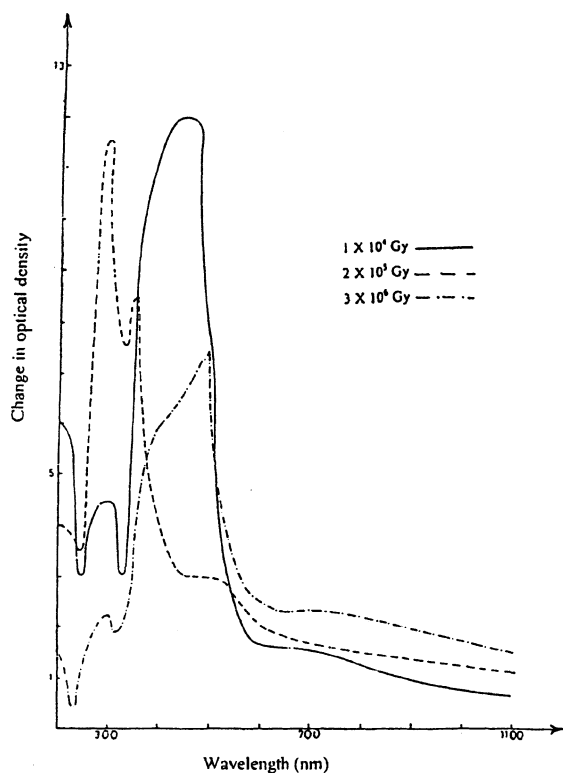


FIG. 13. Induced spectra of the ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, Na₂O 5%, containing 0.05% Cr₂O₃.

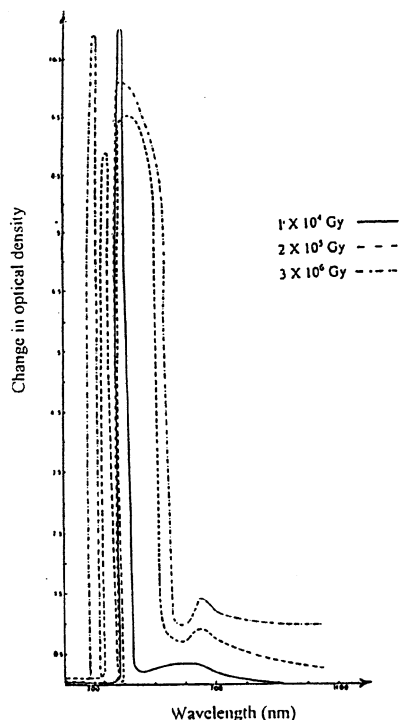


FIG. 14. Induced spectra of the ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, K₂O 5%, containing 0.05% Cr₂O₃.

e. The induced absorption curves (Figs. 12–14), revealed several induced absorption bands at about 300, 370, 420, 600, and 630 nm. The position of these bands changed to longer wavelength with the increase of the irradiation dose; also their intensities increased.

3.3. Effect of the Divalent Metal Oxides

The results obtained from the glass of the above same base composition containing 0.05 g Cr₂O₃/100 g glass in which five parts of lead oxide were replaced by one of the divalent metal oxides, magnesia, zinc oxide, lime, strontium oxide, or barium oxide, cation for cation (Table 1), are shown in Figs 15–19, from which the following can be seen.

a. The absorption bands were comparable and similar to the characteristic absorption bands observed for binary lead borate glasses. The intensity of these bands slightly changed with the introduction of any of the various studied divalent metal oxides.

b. On exposure to gamma rays, the following variations were observed:

1. the intensity of the absorption band at ≈ 650 nm gradually increased with the increase of the gamma radiation, while its position was not changed; and

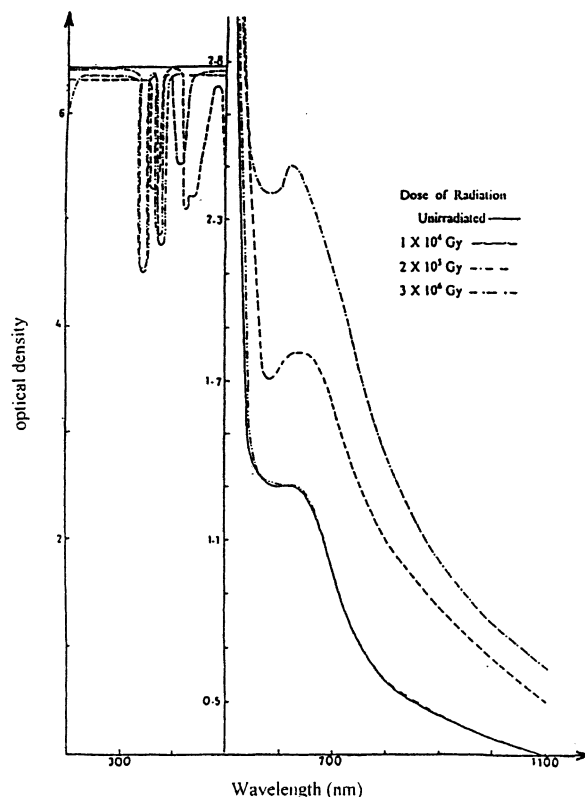


FIG. 15. Absorption spectra of the gamma-irradiated ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, MgO 5%, containing 0.05% Cr₂O₃.

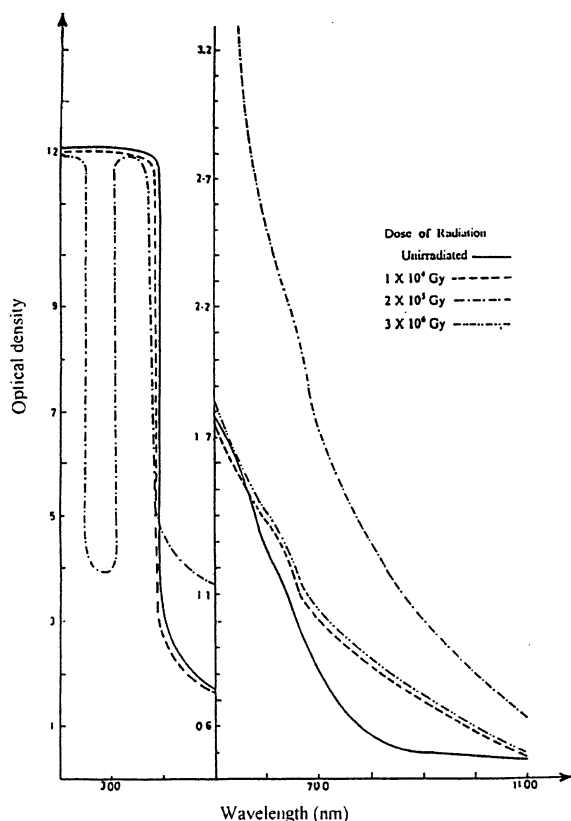


FIG. 16. Absorption spectra of the gamma-irradiated ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, ZnO 5%, containing 0.05% Cr₂O₃.

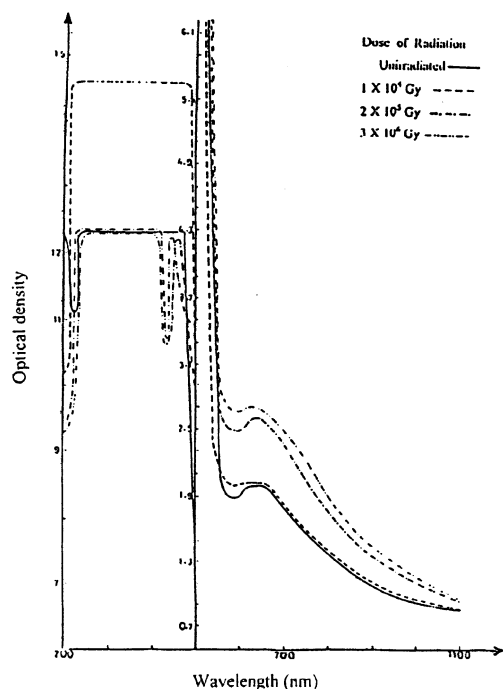


FIG. 17. Absorption spectra of the gamma-irradiated ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, CaO 5%, containing 0.05% Cr₂O₃.

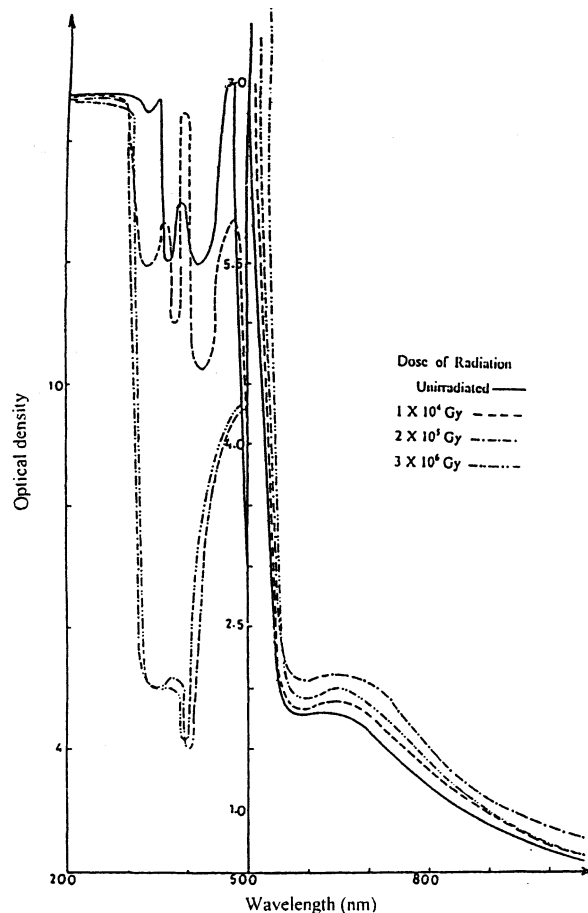


FIG. 18. Absorption spectra of the gamma-irradiated ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, SrO 5%, containing 0.05% Cr₂O₃.

2. the cutoff absorption extending from 200 to 350 nm, either did not change or showed a decrease and sometimes revealed splitting to several absorption peaks.
- c. The induced absorption curves (Figs. 20–24) mostly revealed four absorption bands at ≈ 370 , 430, 520, and 650 nm. The position of these bands was not changed while its intensity increased with the increase of the irradiation dose. The intensity of the extended induced absorption bands at 300–500 nm abnormally increased, exhibiting combination to the composite band with three identified peaks.

3.4. Effect of the Trivalent Metal Oxide

The ternary lead borate glasses containing alumina were studied by replacing parts of boric oxide by alumina, cation for cation (Table 1), and the results obtained revealed the following.

- a. The resolution and the intensity of the visible absorption bands at ≈ 420 and 650 nm were observed to become prominent with the increase of the alumina content. The

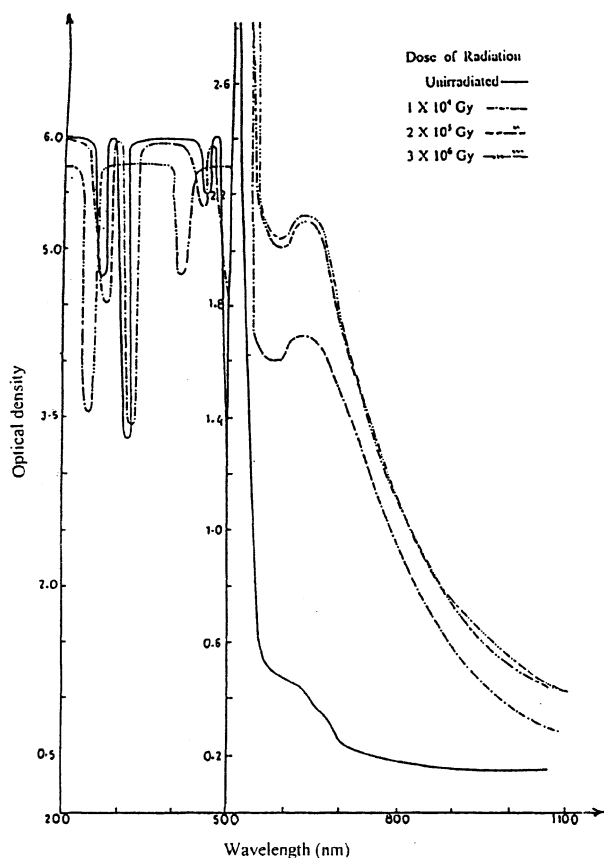


FIG. 19. Absorption spectra of the gamma-irradiated ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, BaO 5%, containing 0.05% Cr₂O₃.

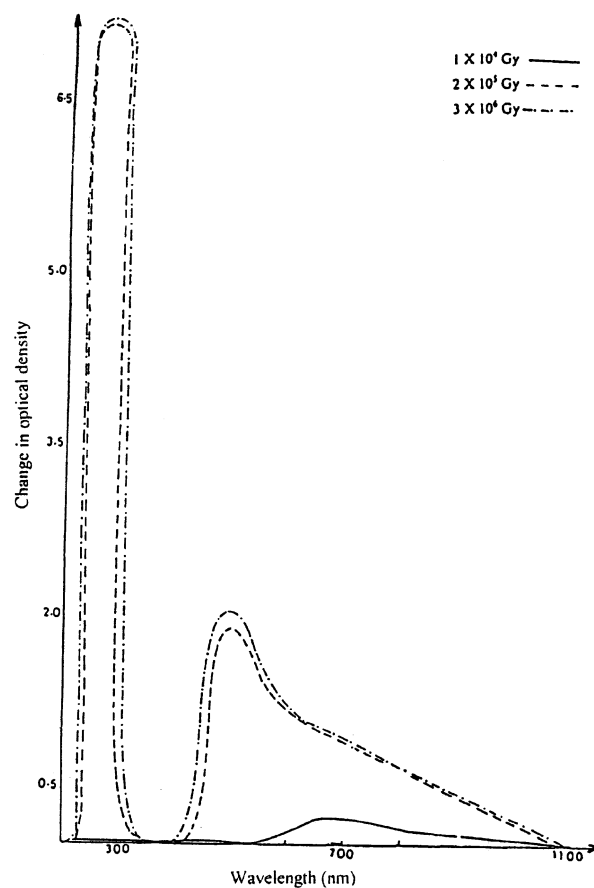


FIG. 21. Induced spectra of the ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, ZnO 5%, containing 0.05% Cr₂O₃.

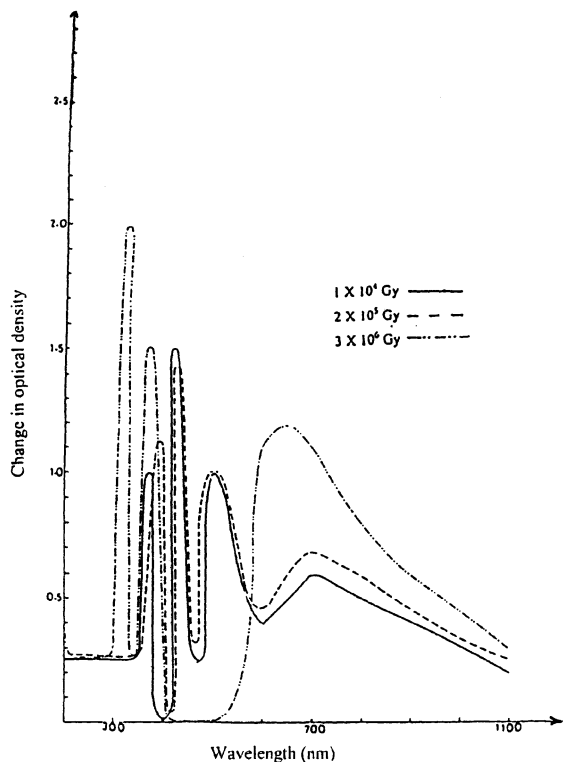


FIG. 20. Induced spectra of the ternary lead borate glass of the composition PbO 75%, B₂O₃ 20%, MgO 5%, containing 0.05% Cr₂O₃.

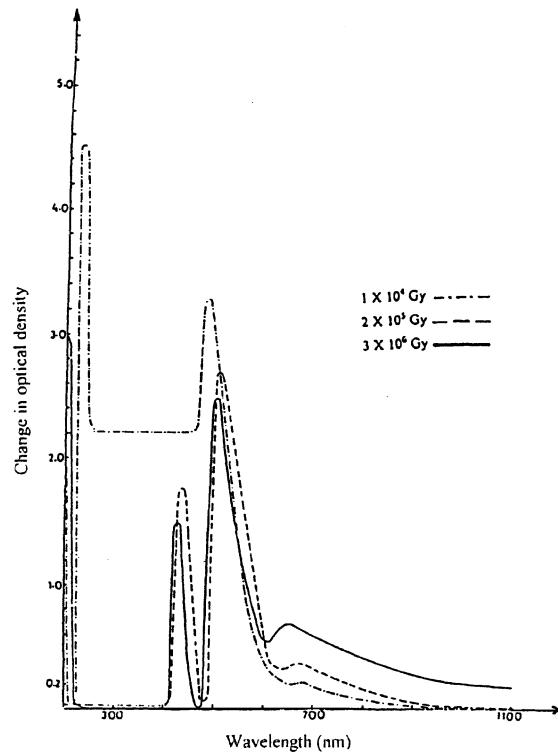


FIG. 22. Induced spectra of the ternary lead borate glass of the composition PbO 75%, B₂O₃ 50%, CaO 5%, containing 0.05% Cr₂O₃.

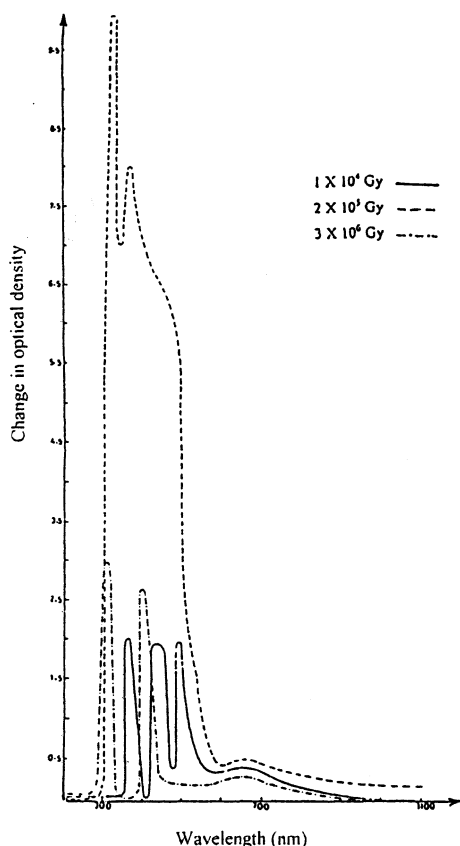


FIG. 23. Induced spectra of the ternary lead borate glasses of composition PbO 75%, B₂O₃ 20%, SrO 5%, containing 0.05% Cr₂O₃.

absorption bands in the ultraviolet region of the spectrum were almost consistent.

b. On the progressive irradiation dose, the intensity of the absorption band at ≈ 650 nm increased with the radiation while the second visible absorption band at ≈ 420 nm and the ultraviolet absorption spectra were not changed or in some instances were splitted to several peaks (Figs. 25 and 26).

c. The induced absorption curves (Figs. 27 and 28) revealed several induced absorption bands at both the visible and the ultraviolet regions of the spectrum. The intensity of these induced absorption bands increased with the increase of the irradiation dose.

4. GROWTH BEHAVIOR OF SOME SELECTED GLASSES

The growth of the absorption band at 650 nm of some glasses with radiation was studied. The results obtained are shown in Figs. 29–31, from which it can be seen that the increasing rate of this absorption band on subjecting these glasses to successive gamma-ray doses was observed to be fast at first, and then a slower rate was followed and a saturation or equilibrium state was approached.

5. DISCUSSION

Chromium normally exists in the glass in two different forms: trivalent and hexavalent. From the similarity of the absorption spectra of the trivalent chromium ion in the glass, aqueous solutions, and crystals, it has an average coordination number of six in the glass which has two characteristic absorption bands at 410–430 and 600–675 nm, originating from the electron transfer with the chromium ion itself (12). The hexavalent chromium ion forms tetrahedral CrO₄²⁻ groups and usually has an ultraviolet absorption band at about 360 nm, originating from the electron transfer from the chromium ion to the coordinated neighboring oxygens.

Tables 2 and 3 show the transitions of the absorption spectra of the chromium ion and the induced absorption bands, respectively (8–11, 13).

When the experimental results obtained from all the glasses studied were compared with the energy diagram for d^3 and d^0 systems, in which Cr³⁺ and Cr⁶⁺ ions, respectively, tend to exist (12), it can be concluded that the visible absorption bands at ≈ 420 nm and 630–690 nm would be expected to be associated with Cr³⁺ ions in the octahedral coordination, while the ultraviolet absorption band at ≈ 350 –380 nm would be assumed to arise from Cr⁶⁺ ions. The increase in the intensity of the absorp-

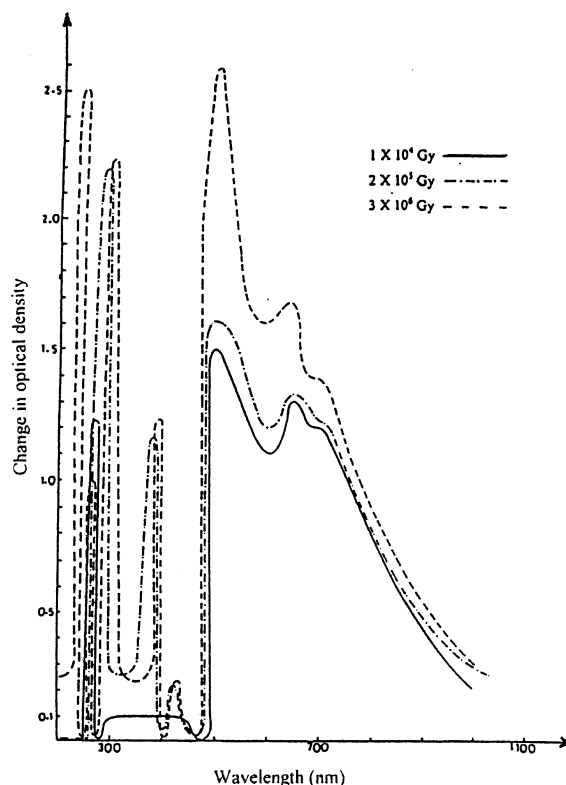


FIG. 24. Induced spectra of the ternary lead borate glasses of composition PbO 75%, B₂O₃ 20%, BaO 5%, containing 0.05% Cr₂O₃.

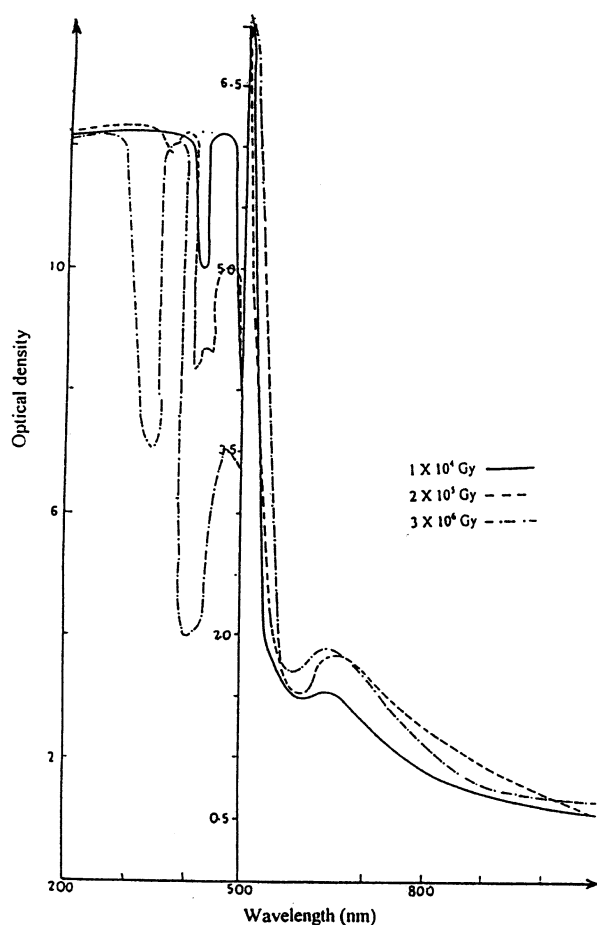


FIG. 25. Absorption spectra of the gamma-irradiated ternary lead borate glass of the composition PbO 80%, B₂O₃ 18%, Al₂O₃ 2%, containing 0.05% Cr₂O₃.

tion may be attributed to the change of Cr³⁺–Cr⁶⁺ ratios or the state of equilibrium between them. The absorption bands at 290 and 330 nm may be due to the lead ion itself (PbO²⁺).

6. EFFECT OF THE GLASS COMPOSITION

It is generally accepted that the alkali or the most alkaline earth cations are incorporated in the glass as the network-modifiers in the interstitial positions throughout the internal structure arrangement of the glass (14). Bishay (1) assumed that the introduction of the alkali oxide in the glasses containing a transition metal generally simplifies the spectrum and increases the intensity of the *d*–*d* transitions. Also, it was recognized that (15) the introducing and/or increasing the alkali or the most alkaline earth cations, generally increases the high valence state of the transition metal cations. In other words, the alkali and/or the alkaline earth cations shift the Cr⁶⁺–Cr³⁺ equilibrium ratio toward the hexavalent chromium state which normally exists as

CrO₄^{2–} groups with possible polymerization as polychromates (16). In the meantime, the trivalent metal cations are believed to share in the formation of the structural building units as glass formers. Specifically, Al³⁺ cations can act as glass formers through the acceptance of the necessary surplus oxygens provided by the alkali or the divalent metal oxides to form AlO₄ groups. Also, the formation of the more stable tetrahedral BO₄ units from the triangular BO₃ groups necessitates the sharing of the nearby alkali or alkaline earth oxides to satisfy this requirement. The formation of such new building units will necessarily affect the Cr⁶⁺–Cr³⁺ equilibrium in the glass. Thus, in such a situation, the formation of such groups as AlO₄ or BO₄ will shift this equilibrium toward increasing the lower valence Cr³⁺ state.

In the high-lead glass studied, it is evident that the lead oxide constitutes the major chemical constituent of all the glasses and in most glasses, its percentage approaches 75–80 wt%. It is expected, therefore, that the lead oxide would play the most effective factor in the property investigated. The replacement of five parts of lead oxide, cation for cation, by one of the alkaline earth oxides leads to some minor effects. The same holds for the substitution of five parts of boric oxide by the trivalent metal oxide. The

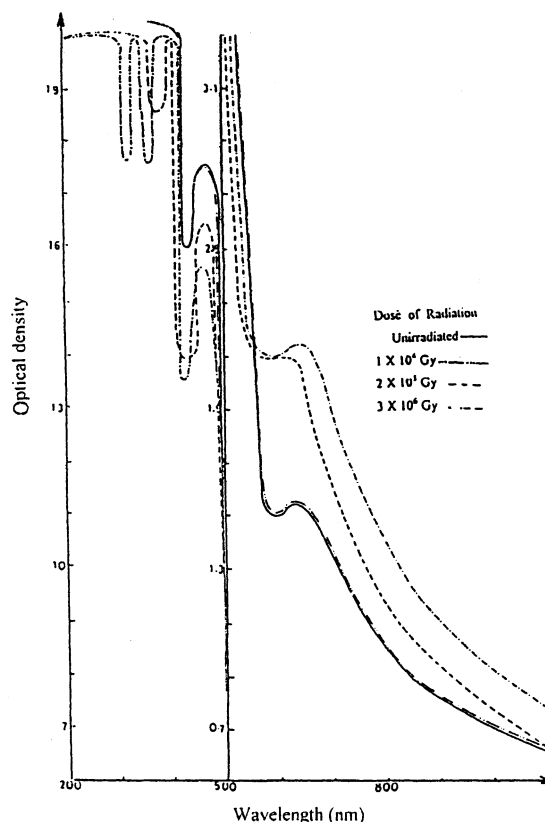


FIG. 26. Absorption spectra of the gamma-irradiated ternary lead borate glass of the composition PbO 80%, B₂O₃ 15%, Al₂O₃ 5%, containing 0.05% Cr₂O₃.

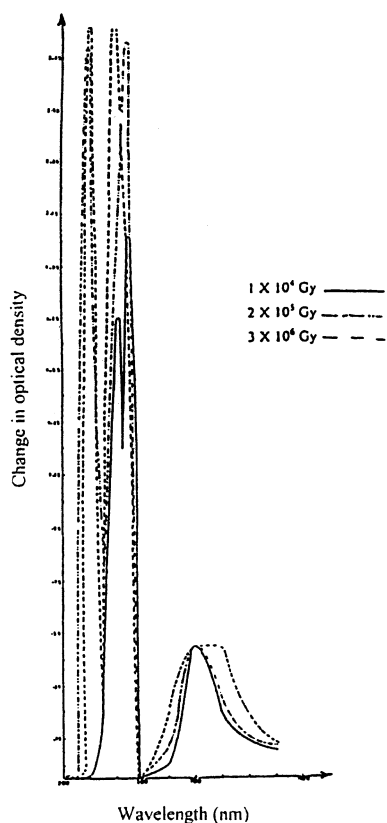


FIG. 27. Induced spectra of the ternary lead borate glass of the composition PbO 80%, B₂O₃ 18%, Al₂O₃ 2%, containing 0.05% Cr₂O₃.

positions of few absorption bands are observed to be slightly changed. No new extra absorption bands or drastic alterations were identified.

On the basis of the previous considerations, the experimental results obtained, on changing the composition of the glass, can be explained as follows:

1. The absorption spectra produced by the transition metal ion are affected by the electric field which the transition metal ion experiences due to the surrounding oxygen ions (the ligand field).

2. The change in the position of the maxima of the absorption bands with the change in the chemical composition could be generally correlated with the ionic radii of the cations present.

3. According to the concept of screening effect (17), the positive fields due the alkali ions are not completely screened by the surrounding oxygen ions. As a result, the effect negative charge of the oxygen ions on the coloring transition metal decreases; the larger the radius of the alkali ions the less perfect the screening and hence the smaller the effective charge. These concepts may give an explanation for the shift of the ligand field energy toward higher energy as one proceeds from K⁺, Na⁺ to Li⁺ or from Ba²⁺, Sr²⁺ to Ca²⁺ ions.

4. The enhanced intensity of the absorption bands which are characteristic for Cr³⁺ ions may indicate greater distortion of the already distorted Cr³⁺ ions which produces more asymmetry and shifts the absorption band maximum further from the centro-symmetric groups, producing a greater intensity in the *d-d* band.

5. The absorption band at about 620–670 nm which is characteristic for Cr³⁺ ion can be hardly seen in the potash containing glass and reveals an increase in resolution and intensity on going from a soda glass to a lithia glass. This can be understood when it is realized that the hexavalent chromium ion increases from Li⁺, Na⁺ to K⁺ ion.

6. The effect of the main building groups such as BO₃ and BO₄ beside the other replaced monovalent, divalent, or trivalent metal oxides is limited on the absorption spectra.

7. EFFECT OF THE GAMMA IRRADIATION

The results obtained from the gamma-irradiated glasses containing chromium revealed that the major effects of the gamma-ray radiation are focused on the progressive increase in the intensity of the visible absorption bands, while

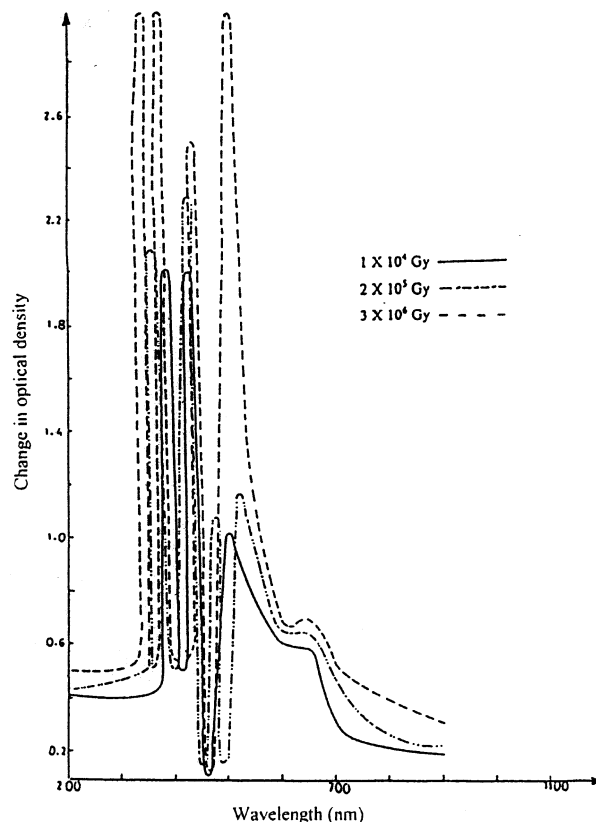


FIG. 28. Induced spectra of the ternary lead borate glass of the composition PbO 80%, B₂O₃ 15%, Al₂O₃ 5%, containing 0.05% Cr₂O₃.

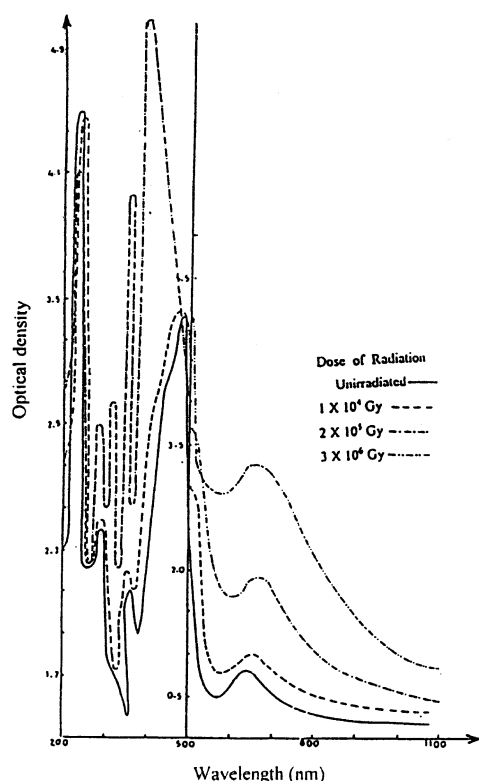


FIG. 29. Absorption spectra of gamma-irradiated binary lead silicate glass of the composition PbO 65%, SiO₂ 35%, containing 0.05% Cr₂O₃.

the ultraviolet bands show sometimes a slight decrease or remain unaffected. Therefore, it seems logical to assume that the observed changes with radiation can be related to one or more of the following.

1. The possibility of the formation and accumulation of new induced defects in the parent host glass on irradiation and hence the production of the characteristic induced color centers of such particular glass system. The introduction of any of the mono-, di-, or trivalent metal oxides investigated seems to have some profound effect on the absorption spectra which can attributed to the change in the Cr³⁺–Cr⁶⁺ ratio by the effect of the polarizable oxygens.

Lead glasses containing appreciable lead oxide content are known to have weakened glass structure; such glass structure is likely to be easily affected by radiation. Induced defects created from the parent glasses are indeed numerous and might suppress further effect due to the presence of chromium.

2. The possibility that lead shares in the formation of new induced color centers by radiation. Apparently, Pb²⁺ can act as both a hole trap, forming Pb³⁺, and an electron trap, forming Pb⁺.

3. The possibility of the sharing of chromium in the formation of new color centers by radiation. In this case the

change in the intensity of the absorption bands can be explained by assuming that some Cr⁶⁺ ions may capture excited electrons and are converted to Cr³⁺.

4. The possibility of the overlapping of the induced bands due to the various species of boric, lead, and chromium oxides.

5. The possibility of the change in the state of equilibrium between Cr³⁺ and Cr⁶⁺ and their relative abundance which vary with many factors.

The above conclusion can be considered by assuming that (18) the rate at which defects and impurity atoms are converted to color centers should be about the same for both. Moreover, the number of the induced color centers formed by impurity must be limited to the number present in the glass and, once all of the specific impurity has been converted, the absorption created should reach a fixed saturation value.

If ionizing radiation causes new defects to be formed, this will be an inefficient process in most instances and the total number of defects will increase very slowly (11).

In the present results, it was observed that almost all glasses reveal an initial fast growth of formation at first followed by a second slower growth frequently reaching saturation (19). These results can be explained by suggesting that before irradiation the lattice structure of the glasses

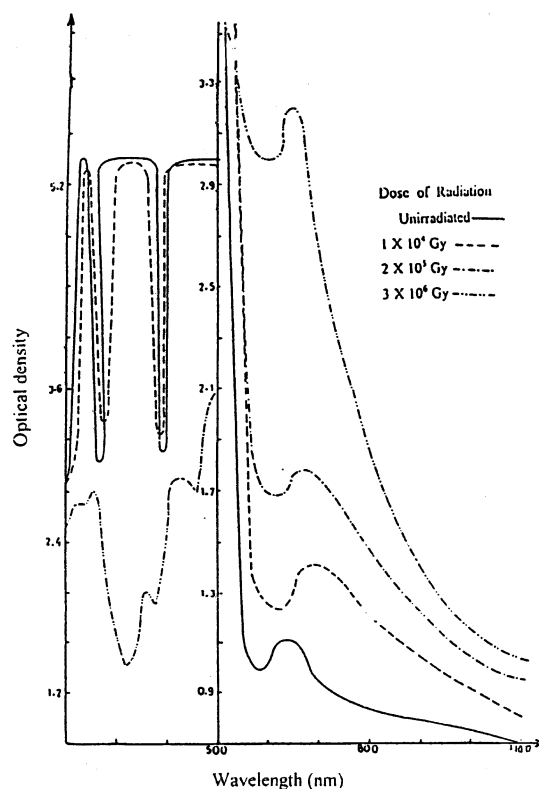


FIG. 30. Absorption spectra of the gamma-irradiated binary lead silicate glass of the composition PbO 80%, SiO₂ 20%, containing 0.05% Cr₂O₃.

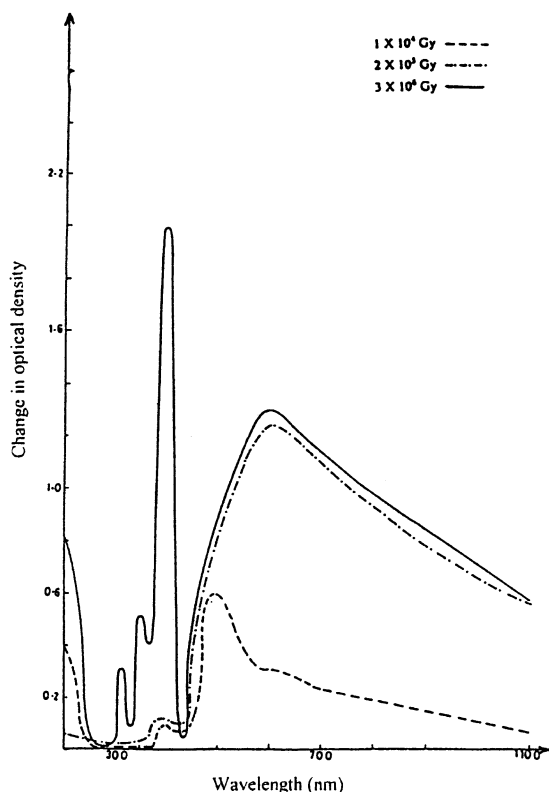


FIG. 31. Induced spectra of the binary lead silicate glass of the composition PbO 65%, SiO₂ 35%, containing 0.05% Cr₂O₃

contains a large number of the intrinsic defects which may trap electrons or positive holes and thus form the color centers, as the glass samples are irradiated. In the meantime, new induced defects may be formed and their number will increase at first with increasing irradiation dose. On the other hand, the number of the intrinsic defects that have not trapped electrons or holes decreases with increasing irradiation. The predominant effect is associated with the intrinsic defects and the rate of formation, accumulation, and annihilation of the color centers associated with these defects.

TABLE 2
Attribution of the Absorption Bands

Position of the absorption bands (nm)	Attribution of the absorption bands
420–450	Cr ³⁺
620–670	
260–290	Cr ⁶⁺
350–380	

TABLE 3
Attribution of the Induced Absorption Bands

Position of the absorption bands (nm)	Attribution of the absorption bands
Ultraviolet	
230–250	Pb center
280–290	Pb center
330	Pb center
350–380	Cr center (Cr ⁶⁺)
Visible	
420	Pb center or Cr center (Cr ³⁺)
480	Pb center
520	Pb center
600–690	Pb center or Cr center (Cr ³⁺)

When the rates of formation and annihilation become nearly equal after prolonged irradiation, an equilibrium or saturation state is assumed to be established (11).

All the above conclusions are in complete agreement with the experimental results obtained.

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