

Research Article

Infrared Spectroscopic Characterization of Tellurite Glasses Containing Heavy Metal Oxides

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The infrared (IR) spectra of $(100-x)\text{TeO}_2-x\text{WO}_3$ glasses reveal that the glass network consists of $[\text{TeO}_3]/[\text{TeO}_{3+1}]$, $[\text{TeO}_4]$, $[\text{WO}_4]$, and $[\text{WO}_6]$ groups as basic structural units. Addition of WO_3 oxide to the binary TeO_2-WO_3 glasses increases the amount of lower coordination of $[\text{TeO}_3]/[\text{TeO}_{3+1}]$ units and decreases the higher coordination of $[\text{TeO}_4]$ units and also the formation of $\text{Te}-\text{O}-\text{W}$ linkages at the expense of $\text{Te}-\text{O}-\text{Te}$ linkages. The IR spectra of $60\text{TeO}_2-(40-x)\text{WO}_3-x\text{PbO}$ glasses reveal that the glass network consists of $[\text{TeO}_3]$, $[\text{TeO}_4]$, $[\text{WO}_4]$, $[\text{WO}_6]$, and $[\text{PbO}_4]$ units. Changes in the coordination state of tellurium and tungsten ions occur when the PbO and WO_3 concentrations are varied. The dual role of the lead ions is confirmed in $60\text{TeO}_2-(40-x)\text{WO}_3-x\text{PbO}$ glass system. The W ion coordination state changes from 4 to 6 when WO_3 concentration increases beyond 30 mol% in both $(100-x)\text{TeO}_2-x\text{WO}_3$ and $60\text{TeO}_2-(40-x)\text{WO}_3-x\text{PbO}$ glass systems. The IR spectra of $60\text{B}_2\text{O}_3-10\text{TeO}_2-(30-x)\text{ZnO}-x\text{PbO}$ glasses reveal that the glass network consists of $[\text{TeO}_3]$, $[\text{BO}_3]$, and $[\text{BO}_4]$ groups.

1. Introduction

Glasses with heavy metal oxides (TeO_2 , GeO_2 , Bi_2O_3 , WO_3 , PbO , Ag_2O , etc.) are promising materials for IR technologies, nonlinear optics, and design of laser devices [1]. Tellurite-based glasses are the subject of intense current research because of the interesting electrical and optical properties. Main features include extended Infrared transmittance [2], high nonlinear optical indices [3], low fusion temperature, and they constitute an excellent matrix for active element doping, justifying a continuous technological interest [4]. The synthesis of glasses with high refractive index values is of great importance in the glass science and the optical industries. Among tellurite glasses, the glasses based on WO_3 , PbO , and other heavy-metal oxides (HMO) are known to have high linear refractive indices ($n > 2.1$) [5–7]. The high linear refractive index of Te^{+4} containing glasses is attributed to the nonbonding lone electron pair $5s^2$ of tellurium [8].

The PbO is unique in its influence on the glass structure and is widely used in glasses because it enhances the resistance against devitrification, improves the chemical durability, and lowers the melting temperature [9, 10]. PbO could act both as glass network former and as modifier

depending on its concentration in the glasses [11, 12]. B_2O_3 is one of the best and well-known glass former. Addition of small amount of TeO_2 into the borate glass network enhances the glass quality with an improvement in transparency, refractive index. Addition of ZnO into the boro-tellurite glass network produces low rates of crystallization and increases the glass-forming ability [13]. Hence it is of interest to study the structural changes brought about by PbO in tungsto-tellurite and boro-tellurite glasses, which may help in predicting the physico-chemical properties.

The aim of the present work is to study the short range structure and structural changes with compositions of $(100-x)\text{TeO}_2-x\text{WO}_3$, $60\text{TeO}_2-(40-x)\text{WO}_3-x\text{PbO}$, and $60\text{B}_2\text{O}_3-10\text{TeO}_2-(30-x)\text{ZnO}-x\text{PbO}$ glass systems by Infrared spectroscopy.

2. Experimental

The tellurium-based glasses $(100-x)\text{TeO}_2-x\text{WO}_3$, $60\text{TeO}_2-(40-x)\text{WO}_3-x\text{PbO}$, and $60\text{B}_2\text{O}_3-10\text{TeO}_2-(30-x)\text{ZnO}-x\text{PbO}$ were prepared from 99.9% purity-grade oxides (Aldrich). Powders of TeO_2 , WO_3 , B_2O_3 , ZnO , and PbO were weighted to get the required composition and ground in a mortar

TABLE 1: Glass compositions.

Samples	Oxides (mol%)				
	TeO ₂	WO ₃	PbO	B ₂ O ₃	ZnO
TW1	90	10	—	—	—
TW2	80	20	—	—	—
TW3	70	30	—	—	—
TW4	60	40	—	—	—
TWP1	60	40	0	—	—
TWP2	60	30	10	—	—
TWP3	60	20	20	—	—
TWP4	60	10	30	—	—
TWP5	60	0	40	—	—
BTZP1	10	—	0	60	30
BTZP2	10	—	10	60	20
BTZP3	10	—	20	60	10
BTZP4	10	—	30	60	0

with a pestle for 1 hour to obtain homogeneous mixtures. Each batch was then transferred to a platinum crucible and melted at about 800–950°C in an electric furnace. This melt was held at this temperature for 30 minutes until a bubble-free liquid was formed. The melts were stirred to achieve desirable homogeneity. The homogeneous melt was quenched by pouring it on to a preheated stainless steel mould to avoid excess thermal shocks. The glasses were annealed for 8 hours at 100°C to relieve the mechanical strains. The compositions of the glass samples employed in the present study are given in Table 1.

X-ray diffractograms of powdered glass samples were recorded using a copper target ($\lambda(k_\alpha) = 1.54 \text{ \AA}^\circ$) on a Philips PW (1140) diffractometer at room temperature. The IR spectra of the glass samples were recorded at room temperature using a Perkin-Elmer FT-IR spectrometer model 1605 using KBr disc technique. The investigated samples were ground to fine particles and then mixed with KBr in the ratio (0.002 : 0.2 g) glass to KBr, respectively. The weighted mixture was then subjected to a pressure of 5 tons/cm². The transmission spectra were measured immediately after preparing the desired disks.

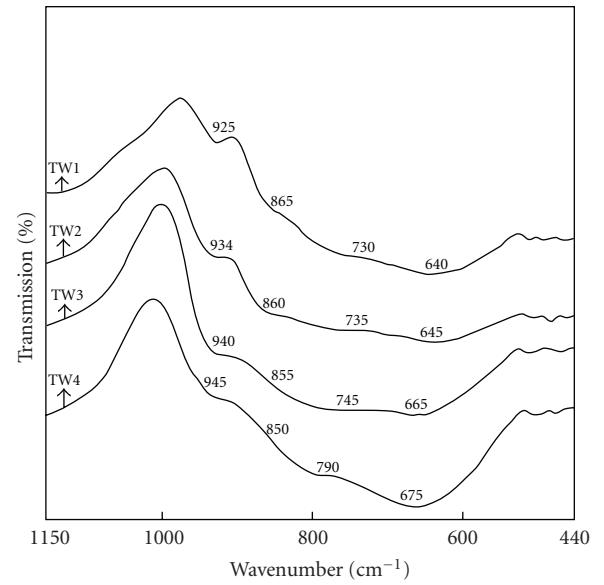
3. Results and Discussion

3.1. XRD and IR Spectra of (100-x)TeO₂-xWO₃ Glasses. The X-ray diffraction spectra show no peaks, indicating that the samples are amorphous.

The IR spectra of (100-x)TeO₂-xWO₃ glass system recorded in the wave number region 1150–440 cm⁻¹ are shown in Figure 1. The IR spectra of these glasses are characterized by IR absorption bands in the wave number regions 925–945 cm⁻¹, 850–865 cm⁻¹, 730–790 cm⁻¹, 600–680 cm⁻¹, and 460–490 cm⁻¹. The IR band positions are summarized in Table 2. The IR absorption in the range 600–680 cm⁻¹ in the tellurium containing glasses is due to stretching vibrations of the Te–O bond in the TeO₄ tbp (trigonal bipyramids) and TeO₃ tp (trigonal pyramids) units [14]. The band observed at 640 cm⁻¹ in 90TeO₂-10WO₃

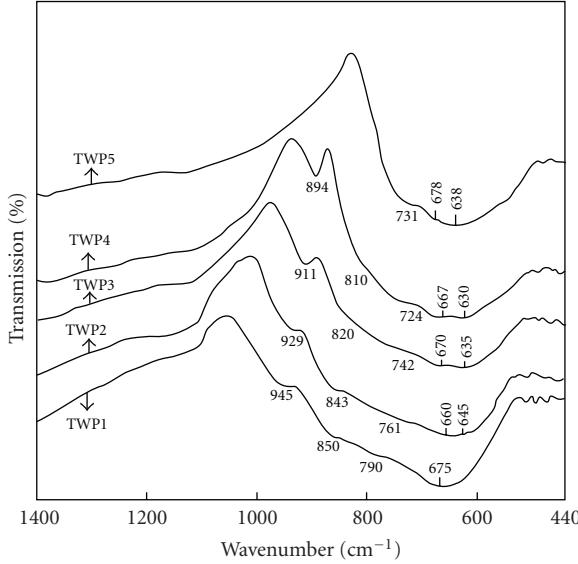
TABLE 2: Observed IR band positions in the (100-x)TeO₂-xWO₃ glass system.

Sample ID	IR bands (cm ⁻¹)					
	460–490	640	—	730	865	925
TW1	460–490	640	—	730	865	925
TW2	460–490	645	—	735	860	934
TW3	460–490	—	665	745	855	940
TW4	460–490	—	675	790	850	945

FIGURE 1: IR spectra of TeO₂-WO₃-glasses.

(TW1) glass is assigned to TeO₄ tbp units. The band at around 925 cm⁻¹ in TW1 in the high wave number region of the spectrum is characteristic of the presence of the tungsten ions in [WO₄] units or [WO₆] units in the structure of the glass [15–17]. The band at around 730–790 cm⁻¹ is due to Te–O_{eq} bond vibrations of distorted TeO₄ units [17, 18]. The shoulder at 865 cm⁻¹ is assigned to vibration of W–O–W linkages [19, 20]. The IR band in the region 460–490 cm⁻¹ is assigned to Te–O–W linkages, which would increase the glass network connectivity and this assignment is made in agreement with the theoretical model for vibrations of mixed bridge bonds containing heavy metal and glass former atoms [21]. The formation of Te–O–W linkages is expected because both W and Te atoms have comparable electro negativity and can therefore substitute for each other in bonding with O atoms [21]. That is, there is a fraction of W cations that have partial covalent bonding and are incorporated in the binary TeO₂-WO₃ glass network [17].

Equimolecular substitution of WO₃ for TeO₂ causes changes in the structure of the TeO₂-WO₃ glasses, which is apparent in the IR spectra of glasses (Figure 1). As the WO₃ content increases from 10 mol% to 40 mol%, the band at 640 cm⁻¹ in 90TeO₂-10WO₃ (TW1) shifts towards higher wave numbers: from 640 to 645, 665, and then to 675 cm⁻¹, while the band at 730 cm⁻¹ shifts to 790 cm⁻¹. This observed shift may be due to higher field intensity of

FIGURE 2: IR spectra of $\text{TeO}_2\text{-}\text{WO}_3\text{-PbO}$ glasses.

mixed $\text{Te}-\text{O}-\text{W}$ and $\text{W}-\text{O}-\text{W}$ linkages, in which the oxygen is highly polarized, compared with $\text{Te}-\text{O}-\text{Te}$ linkages, since W^{6+} -ions possess higher field intensity than Te^{4+} -ions [18], and this observed shift may be related to the apparition of TeO_3 units concomitant to a reduction in the number of TeO_4 units [22]. Thus the effect of addition of WO_3 content to the TeO_2 glass matrix is to transform the TeO_4 basic structural units forming the TeO_2 glass to TeO_3 units. This is supported by the appearance of IR band in the region 665–680 cm^{-1} of $70\text{TeO}_2\text{-}30\text{WO}_3$ (TWP3) and $60\text{TeO}_2\text{-}40\text{WO}_3$ (TWP4) glass systems corresponding to TeO_3 units.

The shoulder at about 850–865 cm^{-1} relative to the existence of vibration of $\text{W}-\text{O}-\text{W}$ linkages is present for all the glass samples. As the WO_3 content increases from 10 to 40 mol%, this causes a shift in the absorption band at 925 cm^{-1} due to WO_4 units toward higher wave numbers: from 925 to 934, 940, and then to 945 cm^{-1} . The observed shift in the 925 cm^{-1} band toward higher wave number in the composition range from 10 to 40 mol% WO_3 is an indication of the transformation of WO_4 units to WO_6 units. However, the substitution of TeO_2 by WO_3 causes an increase of networking oxygens, according to the proportion 3/2 and these oxygens form new $\text{Te}-\text{O}-\text{W}$ and $\text{W}-\text{O}-\text{W}$ linkages. Thus the glass network is highly distorted and this suggests the formation of WO_6 units at high WO_3 content.

3.2. IR Spectra of $60\text{TeO}_2\text{-}(40-x)\text{WO}_3\text{-}x\text{PbO}$ Glasses. The IR spectra of the ternary $\text{TeO}_2\text{-}\text{WO}_3\text{-PbO}$ (TWP) glass system recorded in the wave number region 1400–440 cm^{-1} are shown in Figure 2. The IR spectra of the glasses are characterized by absorption bands in the wave number regions 890–945 cm^{-1} , 810–850 cm^{-1} , 724–790 cm^{-1} , 600–680 cm^{-1} , and 460–490 cm^{-1} . The IR band positions are summarized in Table 3. The band in the region 670–680 cm^{-1} in $60\text{TeO}_2\text{-}40\text{WO}_3$ (TWP1) glass is assigned to

TABLE 3: Observed IR band positions in the $60\text{TeO}_2\text{-}(40-x)\text{WO}_3\text{-}x\text{PbO}$ glass system.

Sample ID	IR bands (cm^{-1})					
TWP1	460–490	—	675	790	850	945
TWP2	460–490	645	660	761	843	929
TWP3	460–490	635	670	742	820	911
TWP4	460–490	630	667	724	810	894
TWP5	460–490	638	678	731	—	—

TeO_3 trigonal pyramids, while the band at around 724–790 cm^{-1} is due to $\text{Te}-\text{O}_{\text{eq}}$ bond vibrations of distorted TeO_4 units. The shoulder at 945 cm^{-1} in TWP1 in the high wave number region of the spectrum is a characteristic of the presence of tungsten ions in $[\text{WO}_6]$ units in the structure of the glass [17]. The shoulder at around 810–850 cm^{-1} is due to vibration of $\text{W}-\text{O}-\text{W}$ linkages and the band in the region 460–490 cm^{-1} is assigned to $\text{Te}-\text{O}-\text{W}$ linkages [17]. Equimolecular substitution of PbO for WO_3 causes changes in the structure of the TWP glasses, which is apparent in the IR spectra of glasses (Figure 2).

As the PbO content increases from 10 to 30 mol%, this causes a shift in the IR band due to $\text{W}-\text{O}-\text{W}$ linkages towards lower wave numbers: from 850 to 843, 820, and then to 810 cm^{-1} and also causes a shift in the band 945 cm^{-1} due to WO_6 octahedra toward lower wave numbers: from 945 to 929, 911 and then to 894 cm^{-1} . The observed shift in the 945 cm^{-1} band and in the band 850 cm^{-1} toward lower wave number in the composition range from 10 to 30 mol% PbO may be an indication of the transformation of WO_6 units to WO_4 units. This receives support from the appearance of band at 894 cm^{-1} in $60\text{TeO}_2\text{-}30\text{PbO}\text{-}10\text{WO}_3$ (TWP4) and is due to WO_4 units [23]. When WO_3 is substituted mol by mol by PbO the number of oxygens in the glass network diminishes according to the ratio 3/1. Thus the glass network becomes less distorted and this also suggests that the formation of WO_4 units at high PbO content. The shoulder at about 850 cm^{-1} is absent in the IR spectrum of $60\text{TeO}_2\text{-}40\text{PbO}$ glass.

When the PbO content increases from 10 to 20 mol%, a new band at around 635 cm^{-1} is observed in addition to that of ~670 cm^{-1} , which indicates the formation of $\text{Te}-\text{O}-\text{Pb}$ bonds in the glass network and this suggests formation of TeO_4 units at the expense of TeO_3 units. Thus it is concluded that addition of PbO causes a change in the coordination state of the tellurium ions and their partial conversion from 3-coordination to 4-coordination. This is supported by the appearance of band in the 600–645 cm^{-1} region corresponding to $[\text{TeO}_4]$ units. At low proportion of PbO (up to 20 mol%), it enters the glass network by breaking up the $\text{Te}-\text{O}-\text{Te}$, $\text{Te}-\text{O}-\text{W}$ bonds and introduces coordination defects known as dangling bond ($\text{Te}-\text{O}^- \cdots \text{Pb}^{2+} \cdots \text{O}-\text{Te}$) which in turn decreases the TeO_3 units by forming TeO_4 units [24]. When the PbO content is increased from 20 to 40 mol%, a considerable proportion may act as double bridges between adjacent TeO_4 and WO_4 units such as = $\text{Te}-\text{O}-\text{Pb}-\text{O}-\text{W}$ = which can be formed beside the formation of PbO_4 units [25]. The lead in this case acts as a glass forming

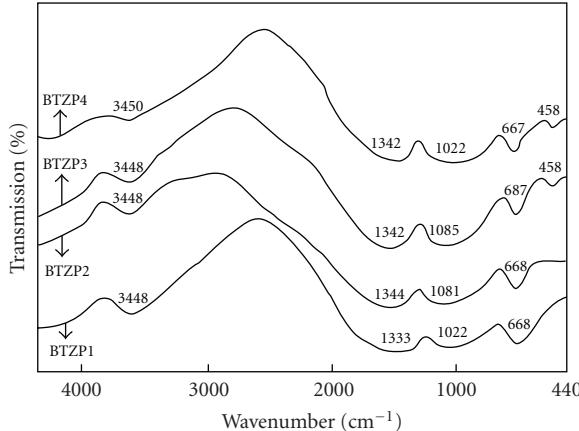


FIGURE 3: IR spectra of B_2O_3 – TeO_2 – ZnO – PbO glasses.

agent and is incorporated in the glass structure in the form of $[\text{PbO}_4]$ units.

Therefore, for PbO up to 20-mol%, it plays a network modifier role, and at $x \geq 30$, it plays a network former role in the present system. The study performed clarified the structural species of lead-tungsten tellurite glasses and confirmed the dual structural role of lead ions.

3.3. IR Spectra of $60\text{B}_2\text{O}_3$ – 10TeO_2 – $(30-x)\text{ZnO}$ – $x\text{PbO}$ Glasses. The IR spectra of the quaternary $60\text{B}_2\text{O}_3$ – 10TeO_2 – $(30-x)\text{ZnO}$ – $x\text{PbO}$ (BTZP) glass system recorded in the wave number region 4000 – 440 cm^{-1} are shown in Figure 3. The IR spectra of these glasses are characterized by absorption bands in the wave number regions 3436 – 3450 cm^{-1} , 1333 – 1390 cm^{-1} , 950 – 1085 cm^{-1} , 665 – 687 cm^{-1} , and 458 cm^{-1} . The IR band positions of the present glass system are summarized in Table 4.

The IR band absorption in the high wave number region of the spectrum with maximum in the 3436 – 3450 cm^{-1} region belongs to O–H stretching vibrations [26]. In the present glass system (BTZP), the disappearance of the absorption band at 806 cm^{-1} indicates the absence of boroxol ring formation and thus clearly indicating the presence of triborate $[\text{BO}_3]$ and tetra borate $[\text{BO}_4]$ units. The IR absorption band in the 950 – 1085 cm^{-1} in all the glasses is due to stretching vibrations of B–O bond of $[\text{BO}_4]$ units [27, 28] and the absorption band in the 1333 – 1390 cm^{-1} region can be attributed to stretching vibrations of B–O bond of $[\text{BO}_3]$ units [29].

The absorption band at around 668 cm^{-1} indicates the presence of tellurium-oxygen groups. The structure pattern of tellurium-containing glasses is determined by trigonal pyramids $[\text{TeO}_3]$ and trigonal bipyramids $[\text{TeO}_4]$. The absorption in the range of 600 – 700 cm^{-1} in such glasses is determined by the stretching vibrations of Te–O bonds in $[\text{TeO}_3]$ and $[\text{TeO}_4]$ units. The absorption of $[\text{TeO}_3]$ units has a high-wave number position than $[\text{TeO}_4]$ units. In general case, the absorption band range of $[\text{TeO}_3]$ units correlates with 650 – 700 cm^{-1} and that of $[\text{TeO}_4]$ units correlates with 600 – 650 cm^{-1} [14, 30]. Analyzing the obtained results and

TABLE 4: Observed IR band positions in the $60\text{B}_2\text{O}_3$ – 10TeO_2 – $(30-x)\text{ZnO}$ – $x\text{PbO}$ glass system.

Sample ID		IR bands (cm^{-1})			
BTZP1	—	668	1022	1333	3448
BTZP2	—	668	1081	1344	3448
BTZP3	458	687	1085	1342	3448
BTZP4	458	667	1022	1342	3450

comparing them to the published data [12], it is clear that trigonal pyramids $[\text{TeO}_3]$ are present in the structure of all the glass systems (BTZP) because the absorption band appears at 667 – 685 cm^{-1} region. For lower concentration of PbO (20 mol%), PbO substituted for ZnO in BTZP series causes changes in the structure of the glasses, which is apparent in a shift of the absorption band at 668 cm^{-1} to higher wave number side up to 687 cm^{-1} . Usually a shift of absorption bands to higher wave numbers occurs as a result of an increase in the degree of polymerization of the structural network $[\text{TeO}_3]$ units of the glass system. Absorption band at 687 cm^{-1} shifted to the lower wave number side, as the mole percentage of PbO increased from 20 to 30. These changes are may be due to formation of Te–O–Pb linkages and dual role of lead ions in the glass network.

In Figure 3, the band around 1022 cm^{-1} in BTZP1 shifts up to 1085 cm^{-1} as the content of PbO increases up to 20 mol% and the band around 1085 cm^{-1} shifts up to 1022 cm^{-1} as the content of PbO increases from 20 to 30 mol%. In lead borate glasses [31], at low concentrations (15–20 mol%) PbO acts as a modifier of the structural network in the form of $[\text{PbO}_6]$ groups and promotes conversion of $[\text{BO}_3]$ units to $[\text{BO}_4]$ tetrahedra. Above 15–20 mol% PbO , some lead ions enter the structure as network former in the form of $[\text{PbO}_4]$ units. The absorption band at 458 cm^{-1} in BTZP3 and BTZP4 probably belongs to the Pb–O vibration of $[\text{PbO}_4]$ units [32]. Thus the fraction of $[\text{BO}_4]$ units increases as the content of PbO increases from 20 to 30 mol%. It is clear from Figure 3 that there is no significant change in position but there is clear evidence of a decrease in the intensity of the absorption band around 1342 cm^{-1} , and a corresponding increase in the intensity of the band at 1022 cm^{-1} is observed in the spectra of all the glasses in BTZP series. This observation suggests that the number of $[\text{BO}_3]$ units decreases and the fraction of $[\text{BO}_4]$ units increases as the PbO content increases. The study performed clarified the structural species of B_2O_3 – TeO_2 – ZnO – PbO glasses and confirmed the dual structural role of lead ions.

4. Conclusions

Transparent and stable glasses were obtained in the TeO_2 – WO_3 , TeO_2 – WO_3 – PbO , and B_2O_3 – TeO_2 – ZnO – PbO glass systems. The IR results show the progressive transformation of the TeO_4 units to more distorted TeO_{3+1} units for TeO_2 – WO_3 glasses with increasing WO_3 content. Addition of PbO and WO_3 increases the population of lower coordination units $[\text{TeO}_3]/[\text{TeO}_{3+1}]$ in the TeO_2 – WO_3 – PbO glass network at the expense of higher coordination units $[\text{TeO}_4]$. Addition

of WO_3 to the glass systems of TeO_2-WO_3 and $\text{TeO}_2-\text{WO}_3-\text{PbO}$ results in the formation of more W–O–W and W–O–Te linkages, while that of the Te–O–Te linkages decreases as WO_3 increases. In the case of $\text{TeO}_2-\text{WO}_3-\text{PbO}$ glasses, the $\text{TeO}_3/\text{TeO}_{3+1}$ units are transformed to TeO_4 units with increasing PbO content. Increasing the PbO content in $\text{B}_2\text{O}_3-\text{TeO}_2-\text{ZnO}-\text{PbO}$ glasses causes partial conversion of the boron ions from trigonal coordination $[\text{BO}_3]$ to $[\text{BO}_4]$ tetrahedral coordination. $[\text{TeO}_3]$ units are present in all the glass samples of $\text{B}_2\text{O}_3-\text{TeO}_2-\text{ZnO}-\text{PbO}$ glass system and the dual role of the lead ions is confirmed in this glass system.

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