A new lead strontium borate (B14O25Pb1.69Sr2.31) and lead barium borate (Pb5.33Ba10.67B44F4O80) have been prepared by crystallizing the corresponding glassy phases of the systems; SrO-B2O3-PbF2 and BaO-B2O3-PbF2 respectively. The glasses were prepared by the melting/quenching technique. The glass crystallization was studied by differential scanning calorimetry (DSC). We observed one exothermic event indicating that crystallization occurs in one steps. Glass samples were heat treated at temperature of crystallization determined by DSC. The crystal structure has been determined by powder X-ray diffraction. B14O25Pb1.69Sr2.31 (isostructural with B14O2Sr4 [1]) crystallizes in the Monoclinic space group C2/m with unit cell a = 16.4274 Å, b = 7.782215 Å, c=16.58075 Å and alfa = gamma and beta = 119.2482 °. Pb5.33Ba10.67B44F4O80 crystallizes in the Orthorhombic space group Cmc21 with unit cell a = 18.78672 Å, b = 10.69704 Å, c= 8.60674 Å and alfa=beta=gamma= 90 ° (isostructural with Ba4B11O20F ) [2]. These crystals were doped with rare earth (Er3+ and Yb3+). As the dopants concentration increases the glass crystallization tendency diminish. The crystal structure determination was very important in the explaining of the difference luminescent properties of the corresponding glass ceramics materials.

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