Orthoborate Halides with the Formula \((M^{III})_5(BO_3)_3X\): Syntheses, Crystal Structures and Raman Spectra of Eu\(_5(BO_3)_3\)Cl and Ba\(_5(BO_3)_3\)X \((X = Cl, Br)\)

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Single crystals of Eu\(_5(BO_3)_3\)Cl were obtained by serendipity by reacting Eu\(_2O_3\) and Mg with B\(_2O_3\) at 1300 K in the presence of an NaCl melt for 13 h in silica-jacketed Nb ampoules. Ba\(_5(BO_3)_3\)X \((X = Cl, Br)\) crystals were formed by direct synthesis from appropriate amounts of Ba(OH)\(_2\), H\(_3BO_3\) and the respective barium halide (hydrate) in alumina crucibles kept in the open atmosphere at 1300 K for 13 h. The crystal structures of the title compounds were determined with single-crystal X-ray diffraction. All compounds crystallize isotypically to Sr\(_5(BO_3)_3\)Cl in the orthorhombic space group C\(222_1\) (no. 20, \(Z = 4\)) with the lattice parameters \(a = 1000.34(7)\), \(b = 1419.00(9)\), \(c = 739.48(5)\) pm for Eu\(_5(BO_3)_3\)Cl, \(a = 1045.49(5)\), \(b = 1487.89(8)\), \(c = 787.01(4)\) pm for Ba\(_5(BO_3)_3\)Cl, and \(a = 1048.76(7)\), \(b = 1481.13(9)\) and \(c = 801.22(5)\) pm for Ba\(_5(BO_3)_3\)Br. The Raman spectra of all compounds were acquired and are presented and compared to literature data. The incremental volume of the orthoborate \((BO_3)_3^{3-}\) anion has been determined and is compared to the Biltz volume.

Key words: Incremental Volume, Barium, Europium, Chloride, Bromide, Orthoborate, Raman Spectra