A dinuclear dysprosium(III) \( p \)-fluorobenzoate 1,10-phenanthroline complex, \([\text{Dy}(p\text{-FBA})_3\text{phen}]_2\) was synthesized and characterized by elemental analysis, UV and IR spectroscopy, single crystal X-ray diffraction, molar conductance, and TG-DTG techniques. It crystallizes in the triclinic space group \( P\bar{1} \) with \( a = 9.895(5), b = 11.754(6), c = 14.756(10) \) Å; \( \alpha = 106.660(9)^\circ, \beta = 107.956(9)^\circ, \gamma = 101.472(7)^\circ; Z = 1. \) The Dy(III) ions are eight coordinate including one terminal bidentate chelating carboxylate group, four bridging carboxylate groups and one 1,10-phenanthroline molecule. The thermal decomposition of \([\text{Dy}(p\text{-FBA})_3\text{phen}]_2\) has been followed by thermal analysis. The lifetime equation at weight-loss of 10\% was deduced as \( \ln \tau = -27.0798 + 19010.2434/T \) by isothermal thermogravimetric analysis.

**Key words:** \( p \)-Fluorobenzoic Acid, Crystal Structure, 1,10-Phenanthroline, Dysprosium Complex, Thermal Analysis