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Synthesis, Spectroscopic Characterization and Crystal Structure of $(EtO)_2P(S)[1\text{-}(NHCH_2)C_6H_4\text{-}3\text{-}(CH_2NH)]P(S)(OEt)_2$

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Bisphosphoramidothioates are widely used as additives in polymers, pesticides in agriculture and drugs in pharmaceutics [1,2]. In this research, we report on the synthesis of $(EtO)_2P(S)[1\text{-}(NHCH_2)C_6H_4\text{-}3\text{-}(CH_2NH)]P(S)(OEt)_2$, belonging to bisphosphoramidothioate family. Characterization was performed by FTIR, NMR (^1H, ^{13}C, and ^{31}P), elemental analysis, Mass spectroscopy and X-ray crystallography. The crystallization solvent was chloroform and n-heptane (4:1 *v*/*v*), and the compound crystallizes in the triclinic system, and space group $P\bar{1}$ with a = 10.0962 (2) Å, b = 10.5568 (2) Å, c = 11.5523 (2) Å, α = 83.6620 (16)°, β = 74.3410 (18)°, γ = 71.0050 (18)°, V = 1120.68 (4) Å^3 and Z = 2. The asymmetric unit consists of one complete molecule. In the crystal structure, the molecules are aggregated through N—H…S═P hydrogen bonds (which appears as red spots on the Hirshfeld surface) in a one-dimensional chain along the *b*-axis. The weaker contacts are C—H…S═P, C—H…π, and CH…HC, manifested as pale red spots on the Hirshfeld surface. The phosphorus signal appears at 72.82 ppm and molecular weight is 441. In the IR spectrum, the band centered at 3275 cm^{-1} is attributed to the NH stretching frequency.

Keywords: Crystal structure, bisphosphoramidothioate, NMR, Synthesis.

References

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