Synthesis, Spectroscopic Study, X-Ray Crystallography Investigation and Hirshfeld Surface Analysis of Three New Phosphorous-Nitrogen Compounds

Mehrdad Pourayoubi*, Anahid Saneei, Banafsheh Vahdani Alviri, Maryam Taherzadeh
Department of Chemistry, Faculty of Sciences, Ferdowsi University of Mashhad, Mashhad, Iran
(e-mail: pourayoubi@um.ac.ir)

New phosphorous-nitrogen compounds 2,3-F_2—$C_6H_3C(O)NHP(O)[N(CH_3)CH_2C_6H_5]_2$ (I), $C_6H_5OP(O)[NHCH(CH_3)_2]_2$ (II) and $(CH_3O)_2P(S)[NHC_6H_4NH]P(S)(OCH_3)_2$ (III) were synthesized and characterized by ^{19}F NMR for (I) and ^{1}H, ^{13}C, ^{31}P NMR and IR and mass spectroscopy and single crystal X-ray determination for all three compounds. In the ^{31}P NMR spectra, the $^{31}P\{^{1}H\}$ signals of (I), (II) and (III) are revealed at 12.95, 10.49 and 69.55 ppm, respectively. The chemical shits observed are within the expected values for analogous phsophoramide and thiophosphoramide compounds.[1,2] In the crystal structure of (I), centrosymmetric dimers are built from pairs of N—H…O=P hydrogen bonds. In the structure (II), the molecules are aggregated in a one-dimensional arrangement *via* N—H…O=P hydrogen bonds along the *b* axis. In the structure (III), the two-dimensional array is generated from the N—H…O hydrogen bonds along the *ab* plane. In structure (II), the oxygen atom of P=O group is a better H-atom acceptor than the oxygen atom of the C_6H_5O group, as the latter oxygen atom doesn't take part in hydrogen bonding interaction. The better hydrogen-bond acceptor capability of P=O group with respect to the oxygen atom of RO group in structures with an $[RO]P(O)[N]_2$ segment were analyzed through the analysis of the structures deposited in the Cambridge Structural Database. In contrast with the structure (II), in the structure (III), the O atom of CH_3O group is involved in N—H….O hydrogen bonding, whereas, the P=S group doesn't take part in the hydrogen bonding interaction (Fig. 1). The Hirshfeld surfaces and fingerprint plots[3] were also employed for analysis of intermolecular interactions.

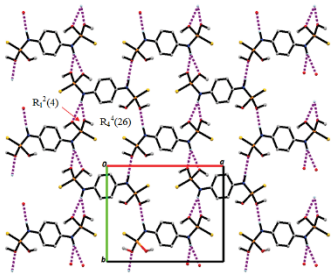

Fig. 1. A crystal packing diagram for structure (III) is represented, showing a 2D network that formed by N-H…O hydrogen bonds.

References

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