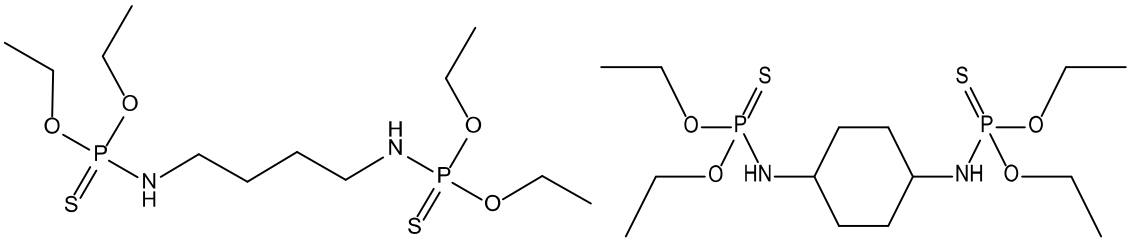
Synthesis and Spectroscopic Characterization of O,O,O',O'-tetraethyl butane-1,4-diylbis(phosphoramidothioate) and O,O,O',O'-tetraethyl cyclohexane-1,4-diylbis(phosphoramidothioate)

Yalda Khoshakhlagh Ostad, Mehrdad Pourayoubi*

Department of Chemistry, Faculty of Science, Ferdowsi University of Mashhad, Mashhad, Iran, y.khoshakhlagh@gmail.com, pourayoubi@um.ac.ir.

Keywords: Bisphosphoramidothioate, NMR spectroscopy, Synthesis.

Structural characteristics have been studied for phosphoramides [1] and thiophosphoramides [2]. These compounds have been attracted attention due to their biological activity and flame retardancy properties [3,4]. In this research, we report the syntheses of $(EtO)_2P(S)[NH(CH_2)_4NH]P(S)(OEt)_2$ (I), and $(EtO)_2P(S)[trans\text{-}1\text{-}(NH)C_6H_{10}\text{-}4\text{-}(NH)]P(S)(OEt)_2$ (II), belonging to bisphosphoramidothioate family, and the characterization by IR, NMR (^1H, ^{13}C, and ^{31}P), elemental analysis, and Mass spectroscopy. In the Mass spectra, the molecular ion peaks are revealed (*m*/*z* = 392 for (I) and 418 for (II)). The phosphorus signals for (I) and (II) appear at 71.68 and 70.33 ppm, respectively. The N—H proton of (I) appears as a singlet at 3.07 ppm, and a similar proton of (II) appears at 3.02. In the IR spectra, the bands centered at 3304 cm^{-1} for (I) and at 3310 cm^{-1} for (II) are attributed to the NH stretching frequencies, and the P=S stretching bands appear at 790 and 787 cm^{-1}, respectively.



Scheme: Chemical structures of (I) (left) and (II) (right).

References:

[1] Pourayoubi, M., Toghraee, M., Divjakovic, V., van der Lee, A., Mancilla Percino, T., Leyva Ramírez, M. A. & Saneei, A. Analysis of N—H· ·O hydrogen bonds in new C(O)-NH-P(O)-based phosphoric triamides and analogous structures deposited in the Cambridge Structural Database. *Acta Cryst.* 2013, *B69*, 184–194.

[2] Pourayoubi, M., Abrishami, M., Eigner, V., Nečas, M., Dušek, M. & Delavar, M. Two new thiophosphoramide structures: N,N',N''-tricyclohexylphosphorothioic triamide and O,O'-diethyl (2-phenylhydrazin-1-yl)thiophosphonate. *Acta Cryst.* 2014, *C70*, 1147–1152.

[3] Levchik, S. V. & Weil, E. D. Flame retardancy of thermoplastic polyesters-a review of the recent literature, *Polym. Int.* 2005, *54*, 11–35.

[4] D. E. C. Corbridge, Phosphorus: chemistry, biochemistry and technology, CRC *press, Boca Raton*.