

Synthesis and spectroscopic characterization of two pairs of phosphoric triamide enantiomers

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Abstract:

Many chiral phosphorus compounds have been synthesized and used in different scientific domains such as medicine [1] and enantioselective catalysts [2], however few number of chiral phosphoric triamides were investigated [3]. In this study, two pairs of enantiomer phosphoric triamides, $\text{CCl}_3\text{C}(\text{O})\text{NHP}(\text{O})[(\text{R})-(+)\text{-NHCH}(\text{CH}_3)(\text{C}_6\text{H}_4\text{-4-CH}_3)]_2/\text{CCl}_3\text{C}(\text{O})\text{NHP}(\text{O})[(\text{S})-(\text{-})\text{-NHCH}(\text{CH}_3)(\text{C}_6\text{H}_4\text{-4-CH}_3)]_2$, denoted (I)/(II), and $\text{CCl}_3\text{C}(\text{O})\text{NHP}(\text{O})[(\text{R})-(+)\text{-NHCH}(\text{C}_2\text{H}_5)(\text{C}_6\text{H}_5)]_2/\text{CCl}_3\text{C}(\text{O})\text{NHP}(\text{O})[(\text{S})-(\text{-})\text{-NHCH}(\text{C}_2\text{H}_5)(\text{C}_6\text{H}_5)]_2$, denoted (III)/(IV), have been synthesized and characterized by melting point, IR, NMR, mass. In the IR spectra of (I)/(II) and (III)/(IV), the bands centered at 3300 cm^{-1} for the first pair and at $3311/3307\text{ cm}^{-1}$ for the second pair are attributed to the NH_{amine} stretching frequencies. The mass spectra, in 70 eV experiments, of (I)/(II) and (III)/(IV) reveal the presence of the molecular ion peaks at $m/z = 477$ (considering one ^{37}Cl and two ^{35}Cl). For (I)/(II) and (III)/(IV), the ^{31}P signals appear at 4.30/4.24 ppm and 4.67/4.75 ppm, respectively. The signals of N_{CPH} , in ^1H NMR spectra, are observed at 9.29, 9.34, 9.55 and 9.42 ppm for (I) to (IV), respectively, all of them to be broad. The two chiral amine fragments in all of the compounds (I), (II), (III) and (IV) reveal two sets of signals in corresponding ^1H and ^{13}C NMR spectra. Typically, the two NH protons in each compound appear as two triplets. The triplet pattern is a result of vicinal H-H and geminal P-H couplings. In the ^{13}C NMR spectra of (I)/(II) and (III)/(IV), the carbon atom of the C(O) group appears as a singlet.

References:

- [1] Gál, B., Bucher, C., Burns, N. Z. (2016). *Mar. Drugs*, **14**, 206–217.
- [2] Lu, Y., Nakatsuji, H., Okumura, Y., Yao, L., Iahihara, K. (2018). *J. Am. Chem. Soc.* **140**, 6039–6043.
- [3] Eghbali Toularoud, M., Pourayoubi, M., Dušek, M., Eigner, V., Damodaran, K. (2018). *Acta Cryst. C* **74**, 608–617.



