ISSN: 10400400
CODEN: STCHE
DOI: 10.1007/s11224-010-9592-z
Document Type: Article
Source Type: Journal

Structural Chemistry
Volume 21, Issue 3, 2010, Pages 629-636

Polymorphism for a novel phosphoramidate; NMR and X-ray crystallography

Gholivand, K.[a], Afshar, F.[b], Shariatinia, Z.[c], Zare, K.[d e]

[a] Department of Chemistry, Tarbiat Modares University, P.O. Box 14115-175, Tehran, Iran
[b] Department of Biology, Islamic Azad University, Garmsar branch, Garmsar, Iran
[c] Department of Chemistry, Amirkabir University of Technology, P. O. Box 159163-4311, Tehran, Iran
[d] Department of Chemistry, Science and Research Branch, Islamic Azad University (IAU), P.O. Box 14515-775, Tehran, Iran
[e] Department of Chemistry, Shahid Beheshty University, Tehran, Iran

Abstract

New phosphoramidates with formula 3-$NC_5H_4C(O)NHP(O)XY$ (X=Y=Cl (1), X=Y=NH-$C(CH_3)_3$ (2a,2b), X=Y=$N(C_4H_9)_2$ (3), X=Cl, Y=$N(C_2H_5)_2$ (4) were synthesized and characterized by IR, ^{1}H-, ^{13}C-, ^{31}P-NMR spectroscopy and CHN elemental analysis. Surprisingly, the reaction of compound 2a with $LaCl_3$, $7H_2O$ in 3:1 M ratio leads to a polymorph of this compound (2b). NMR spectra indicate that $^2J(PNH_{amide})$ in 2b (7.0 Hz) is very much greater than in 2a (4.1 Hz), while $\delta(^{31}P)$ values are identical for both of them. In IR spectra, υ(P=O) is weaker but υ(C=O) is stronger in 2a than in 2b. The structures of 2a, 2b were determined by X-ray crystallography. These compounds form centrosymmetric dimers via two intermolecular P=O..... H-N hydrogen bonds. Strong intermolecular N-H... N, N-H... O and weak C-H... O hydrogen bonds lead to a three-dimensional polymeric cluster in the 2a while intermolecular strong N-H..... N and weak C-H..... O hydrogen bonds form a two-dimensional polymeric chain in 2b. © 2010 Springer Science+Business Media, LLC.

Language of Original Document

English