

The Structural Study of [2-Cl-C₆H₄C(O)NH]P(O)[NHC₆H₄-4-CH₃]₂

Taherzadeh M¹,
Pourayoubi M¹,
Dusek M² and Kucerakova M²

Abstract

A new phosphoric triamide with the formula [2-Cl-C₆H₄C(O)NH]P(O)[NHC₆H₄-4-CH₃]₂ has been investigated by spectroscopic methods and X-ray crystallography. This compound crystallizes in the monoclinic system, with *P*2₁/*c* space group. In this molecule, the P atom has a distorted tetrahedral environment. The N atoms bonded to P atom have mainly sp² character. In the crystal, the molecules are aggregated through N_{cp}-H...O=P and N_p-H...O=C hydrogen bonds in a linear arrangement along the *b* axis, by forming a sequence of alternate R₂²(8) and R₂²(12) motifs (N_{cp} is the nitrogen atom of C(O)NHP(O) segment and the N_p stands the two other nitrogen atoms bonded to the P atom). Furthermore, C-H...O and C-H...Cl intermolecular interactions complete a 3D structure.

Keywords: Phosphoric triamide; Hydrogen bond; Crystal structure; Graph set motif

- 1 Department of Chemistry, Faculty of Sciences, Ferdowsi University of Mashhad, Mashhad, Iran
- 2 Institute of Physics of the Czech Academy of Sciences, Na Slovance 2, 182 21 Prague 8, Czech Republic

Corresponding author: Pourayoubi M

✉ pourayoubi@um.ac.ir

Department of Chemistry, Faculty of Sciences, Ferdowsi University of Mashhad, Mashhad, Iran.

Tel: +989155143058

Received: October 17, 2016; **Accepted:** November 22, 2016; **Published:** November 30, 2016

Introduction

Phosphoramides constitute a well-studied sub-class of phosphorus(V)-nitrogen compounds due to the biological activity of some derivatives and growing applications in pharmacological and agricultural industry [1-3]. These compounds can bind to a metal cation as an oxygen-donor ligand [4-6]. Within this sub-class, compounds with a P(O)[NHC(O)][N]₂ skeleton are interesting for the preparation of chelating bidentate ligands and conformational studies of the C(O)-NH-P(O) fragment [7-12]. Here we report the synthesis, spectroscopic characterizations, and X-ray crystallography of *N*-(2-chlorobenzoyl)-*N'*, *N''*-bis(4-methylphenyl)-phosphoric triamide (C₂₁H₂₁ClN₃O₂P). The possibility of forming different hydrogen bonds is also discussed.

Experimental

X-ray crystallography

A single crystal of the investigated compound was measured with a SuperNova four-circle diffractometer of Rigaku Oxford Diffraction equipped with a 40W micro focus CuKα X-ray source collimated by mirrors, and CCD detector Atlas S2. Measurement was done at 95K using a Cryostream 800 Plus chiller. Measurement and data processing of the CCD images were done by program CrysAlisPro [13], structure was solved by Superflip [14] and refined by Jana 2006 [15]. All atoms except hydrogen

were refined anisotropically. Hydrogen atoms belonging to carbon were kept at the expected positions while positions of hydrogen atoms belonging to nitrogen were refined using a restraint on N-H distances keeping them the same. Isotropic ADP of hydrogen atoms were constrained to 1.2 multiple of the U_{eq} of their parent atom. No unusual features were found during the structure solution and refinement.

Spectroscopic measurements

¹H, ¹³C and ³¹P NMR spectra were recorded on a Bruker Avance III-300 spectrometer. ¹H and ¹³C NMR spectra were referenced using the solvent CDCl₃ resonances (7.29 and 77.07 ppm for ¹H and ¹³C, respectively) for [2-Cl-C₆H₄C(O)NH]P(O)[Cl]₂ and using DMSO-*d*₆ resonances (2.50 ppm, ¹H, and 39.52 ppm, ¹³C) for [2-Cl-C₆H₄C(O)NH]P(O)[NHC₆H₄-4-CH₃]₂. The ³¹P NMR spectra were calibrated using the "absolute referencing" from the related ¹H spectra. IR spectra were recorded on a Thermo Nicolet Avatar 370 FTIR spectrometer and on a Buck 500 scientific spectrometer using KBr discs. The mass spectra were recorded with an MS model of the CH7A Varian Detector. Elemental analyses (C, H

and N) were performed on a Thermo Finnigan Flash 1112EA elemental analyzer and the melting points were recorded with an Electrothermal IA 9000 apparatus.

Synthesis

Caution: Phosphorus pentachloride is very sensitive to the moisture and its partial hydrolysis gives phosphoryl trichloride, so, for the preparation of the $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]\text{P(O)}[\text{Cl}]_2$ reagent, dry CCl_4 solvent was used. Carbon tetrachloride was dried over P_2O_5 under reflux condition and distilled prior to use. A previous article mentioned the synthesis of $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]\text{P(O)}[\text{Cl}]_2$ reagent and its melting point ($92\text{--}93^\circ\text{C}$) [16]. The procedure reported here is similar to the above-mentioned literature method with a few modifications. The modifications consist in replacing the dry C_6H_6 solvent by dry CCl_4 and also using PCl_5 as phosphorus-chlorine reagent instead of $2\text{-Cl-C}_6\text{H}_4\text{C(O)N}=\text{PCl}_3$. A schematic protocol for the synthesis of $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]\text{P(O)}[\text{Cl}]_2$ reagent and $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]\text{P(O)}[\text{NHC}_6\text{H}_4\text{-4-CH}_3]_2$ phosphoric triamide is given in **Scheme 1**. It should be noted that the $2\text{-Cl-C}_6\text{H}_4\text{C(O)N}=\text{PCl}_3$ reagent is also formed during the procedure used in this paper when PCl_5 and $2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}_2$ react (stage (i) in Scheme); however, to avoid the hydrolysis of sensitive $2\text{-Cl-C}_6\text{H}_4\text{C(O)N}=\text{PCl}_3$ reagent, we preferred to continue the reaction with adding HCOOH to the solution containing $2\text{-Cl-C}_6\text{H}_4\text{C(O)N}=\text{PCl}_3$ (stage (ii) in Scheme). We further studied the $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]\text{P(O)}[\text{Cl}]_2$ reagent with IR and NMR experiments and mass spectrometry. Moreover, the fusion point measured by us is a few more than that was reported in literature, probably due to different crystallinities of product obtained in different solvents or the more purity of sensitive reagent obtained by us.

Synthesis of $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]\text{P(O)}[\text{Cl}]_2$

The $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]\text{P(O)}[\text{Cl}]_2$ reagent was prepared by reflux of phosphorus pentachloride and 2-chlorobenzamide in equimolar ratio in dry CCl_4 for 3 h (stage (i) in Scheme). The completion of this stage was monitored by stopping of the evolution of gas bubbles in an oil vessel. The reaction mixture was then cooled at an ice-

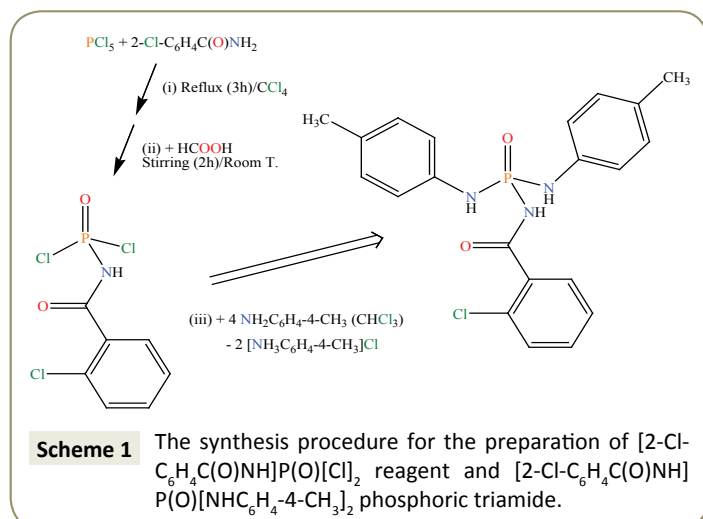
bath and formic acid with similar ratio of the noted reactants was syringed drop-wise into the cold solution, and resulting colorless solution was stirred at 25°C for 2 h. After completion of the reaction, the stirring was stopped and the solvent was removed at a reduced pressure (stage (ii) in Scheme) to form a solid white product (yield: above 80%). Fusion point: 99°C . IR (cm^{-1}): 3091, 2834, 2676, 2553, 1712, 1673, 1592, 1477, 1431, 1405, 1292, 1264, 1227, 1162, 1105, 1046, 960, 901, 873, 776, 752, 716, 653, 592, 561, 509, 465, 405. $^{31}\text{P}\{^1\text{H}\}$ NMR (acetone- d_6): $\delta=1.78$ (s). ^1H NMR (CDCl_3): $\delta=7.41$ (m, 1H), 7.50 (m, 2H), 7.74 (m, 1H), 9.62 (d, $^2\text{J}(\text{P,H})=8.4$ Hz, 1H, NH). ^{13}C NMR (CDCl_3): $\delta=127.34$, 130.51, 130.89, 131.67, 131.80 (d, $^3\text{J}(\text{P,C})=10.4$ Hz), 133.34, 165.64 (d, $^2\text{J}(\text{P,C})=3.6$ Hz). MS (70 eV, EI): m/z (%)=238 (30) $[\text{M}-^{35}\text{Cl}]^+$ (M is based on two ^{35}Cl and one ^{37}Cl), 139 (88) $[\text{C}_7\text{H}_4^{35}\text{ClO}]^+$, 138 (100) $[\text{C}_7\text{H}_5^{35}\text{ClN}]^+$, 101 (63) $[\text{P}^{35}\text{Cl}_2]^+$, 75 (95) $[\text{CH}_2\text{PNO}]^+$, 47 (56) $[\text{PO}]^+$. ($\text{C}_7\text{H}_5\text{Cl}_3\text{NO}_2\text{P}$) (%): C=30.83; H=1.84; N=5.14; found: C=30.83; H=1.72; N=5.21.

Synthesis of $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]\text{P(O)}[\text{NHC}_6\text{H}_4\text{-4-CH}_3]_2$

A solution of p-toluidine (10 mmol) in CHCl_3 (25 ml) was added dropwise to a solution of $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]\text{P(O)}[\text{Cl}]_2$ (2.5 mmol) in the same solvent (25 ml) at 273 K. After 4 h of stirring at an ice bath temperature, the process was stopped and the mixture kept at room temperature for a few days to remove the solvent. The solid obtained was washed with distilled water to remove the $[\text{NH}_3\text{C}_6\text{H}_4(4\text{-CH}_3)]\text{Cl}$ salt (stage (iii) in Scheme). Suitable single crystals were obtained from a solution of the synthesized compound in $\text{CHCl}_3/\text{CH}_3\text{CN}$ (1:4 v/v) at room temperature after a few days (yield: above 80%). Fusion point: 242°C . IR (cm^{-1}): 3336, 3288, 3081, 2889, 1670, 1517, 1446, 1385, 1289, 1216, 945, 821, 740. $^{31}\text{P}\{^1\text{H}\}$ NMR ($\text{DMSO-}d_6$): $\delta=-6.17$ (s). ^1H NMR ($\text{DMSO-}d_6$): $\delta=2.22$ (s, 6H, Me), 7.02 (d, $^3\text{J}(\text{H,H})=8.4$ Hz, 4H), 7.08 (d, $^3\text{J}(\text{H,H})=8.7$ Hz, 4H), 7.36 (m, 2H), 7.45 (m, 2H), 7.74 (d, $^2\text{J}(\text{P,H})=9.9$ Hz, 2H, NH), 10.05 (s, 1H, NH). ^{13}C NMR ($\text{DMSO-}d_6$): $\delta=20.71$, 127.33, 129.78, 131.76, 136.34 (d, $^3\text{J}(\text{P,C})=9.1$ Hz), 138.89, 168.35. MS (70 eV, EI): m/z (%)=415 (8) $[\text{M}]^+$ (^{37}Cl), 413 (40) $[\text{M}]^+$ (^{35}Cl), 412 (43) $[\text{M}-1]^+$, 138 (65) $[\text{C}_7\text{H}_4^{35}\text{Cl}_2\text{CNH}]^+$, 137 (20) $[\text{C}_7\text{H}_4^{35}\text{Cl}_2\text{CNH}]^+$, 107 (50) $[\text{C}_7\text{H}_8\text{NH}]^+$. ($\text{C}_{21}\text{H}_{21}\text{Cl}_2\text{N}_3\text{O}_2\text{P}$) (%): C=60.89; H=5.07; N=10.15; found: C=60.53; H=5.14; N=10.39.

Description of the crystal structure

The asymmetric unit of $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]\text{P(O)}[\text{NHC}_6\text{H}_4\text{-4-CH}_3]_2$ consists of one molecule, as shown in **Figure 1**. The crystal data and refinement parameters are listed in **Table 1** and selected bond distances and angles are listed in **Table 2**. The P atom has a distorted tetrahedral configuration as has been noted for other phosphoric triamides [17], with bond angles around the P atom in the range of $99.54(5)\text{--}118.98(6)^\circ$. All P—N bonds in this compound are shorter than a typical phosphorus-nitrogen single bond (1.77 Å) and longer than a typical phosphorus-nitrogen double bond (1.57 Å) [18], caused probably by the overlap of the electrostatic effects of the P—N polar bonds with their corresponding sigma bonds [19]. The P— N_p distances of 1.6335(11) and 1.6465(11) Å are significantly shorter than the related P— N_{cp} bond distance (1.6862(11) Å), resulting from the electronic effect caused by the C(O) group. This prolongation



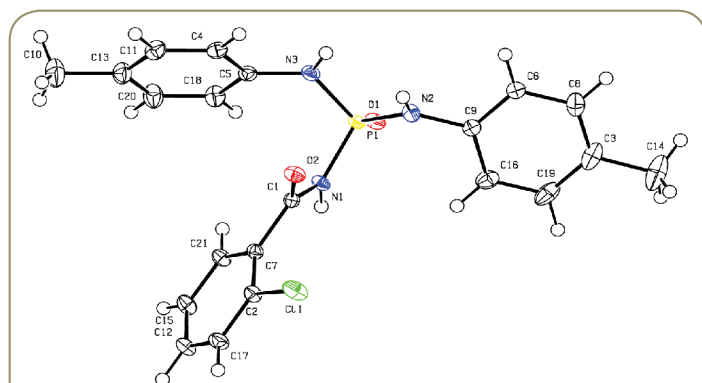


Figure 1 Displacement ellipsoids plot (50% probability) is shown for $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]P(\text{O})[\text{NHC}_6\text{H}_4\text{-4-CH}_3]_2$ with atom numbering scheme. H atoms are drawn as spheres of arbitrary radii.

Table 1. Crystal data and structure refinement for $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]P(\text{O})[\text{NHC}_6\text{H}_4\text{-4-CH}_3]_2$.

Empirical formula	$\text{C}_{21}\text{H}_{21}\text{ClN}_3\text{O}_2\text{P}$
Formula weight	413.8
Temperature (K)	94.9(3)
Wavelength (Å)	1.54184
Crystal system	Monoclinic
Space group	$P2_1/c$
a (Å)	10.3249(2)
b (Å)	9.7374(2)
c (Å)	20.3897(4)
α (°)	90
β (°)	98.1988(17)
γ (°)	90
V (Å ³)	2028.98(7)
Z	4
D_{calc} (g/cm ³)	1.3548
Absorption coefficient (mm ⁻¹)	2.592
$F(000)$	864
Crystal size (mm)	0.15 × 0.105 × 0.063
θ Range for data collection (°)	4.33 to 75.22
Index ranges	$-12 \leq h \leq 12$ $-12 \leq k \leq 12$ $-18 \leq l \leq 25$
Reflections collected	18305
Independent reflections	4133 [$R_{\text{int}} = 0.022$]
Absorption correction	Multi-scan
Max and min transmission	1.000 and 0.918
Refinement method	full-matrix least-squares on F^2
Data/restraints/parameters	4133/2/262
Goodness-of-fit on F^2	1.76
Final R indices [$I > 3\sigma(I)$]	$R_1 = 0.0302$, $wR_2 = 0.0912$
R indices (all data)	$R_1 = 0.0327$, $wR_2 = 0.0933$
Largest difference in peak and hole (e Å ⁻³)	0.34 and -0.28

can also be found in the CSD for different types of P—N bonds in C(O)NHP(O)-based phosphoric triamides [20]. The phosphoryl and carbonyl groups, separated by the NH unit, adopt an anti-

position with respect to each other, which is in agreement with previously reported acyclic phosphoric triamide compounds containing a C(O)NHP(O)(NH)₂ skeleton [21,22]. The P=O bond length is of standard value (1.4770(9) Å), and the bond-angle sums of about 355° for one nitrogen atom and about 360° for two other nitrogen atoms (P—N—C+C—N—H+H—N—P) confirm their sp² character. The criteria for distinguishing between planar and non-planar geometries from bond-angle sums are the same as previously proposed: N(planar) and N(pyramidal) refer to the cases with $\Sigma \geq 352.5^\circ$ and $\Sigma \leq 339.0^\circ$, respectively, and the intermediate entries are the cases with Σ in the range 339.0° – 352.5° [23].

The nitrogen atoms in the title structure do not take part in hydrogen bonding as an acceptor, because they have low Lewis-base character. So, two H-acceptors and three H-donors existing in the structure make three different hydrogen-bonded ring motifs. The more acidic NH of the C(O)NHP(O) moiety (N_{cp}H) participates in an R₂²(8) motif together with P(O), whereas the two other H atoms of the NHR units (N_p) participate in an R₂²(12) motif combined with R₂¹(6) together with C(O). On the other hand, adjacent molecules are linked via a sequence of alternating R₂²(8) and R₂²(12)/R₂¹(6) ring motifs with together C₁¹(4) chain motif in a linear arrangement parallel to b axis (Figure 2). It was found that the strongest N_{cp}—H...O hydrogen bonds exist in the R₂²(8) motif (Table 3), as in recently published papers [24-33]. In addition to the hydrogen bonds noted, there are also the weak C—H...O interactions (between the phosphoryl oxygen with the neighboring aromatic C—H donor of the 2-Cl-C₆H₄ part) through discrete D₁¹(2) hydrogen bond motifs along the a axis. Further stabilization of this compound is achieved via chlorine atom participating in the C—H...Cl hydrogen bond along the c axis (through D₁¹(2) graph-set motifs). These interactions complete a 3D structure of the title compound.

Conclusion

In summary, we reported a new phosphoric triamide, namely, $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}]P(\text{O})[\text{NHC}_6\text{H}_4\text{-4-CH}_3]_2$ that was prepared in good yield and purity by treating 2-chlorobenzoyl phosphoramidic dichloride with *p*-toluidine. It crystallizes in the monoclinic system with $P2_1/c$ space group. Adjacent molecules are linked through (N_p—H...O)=C and (N_{cp}—H...) (C—H...) O=P hydrogen bonds in two-dimensional slabs parallel to ab plane. The slabs consist of chains of alternating R₂²(8) and R₂²(12)/R₂¹(6) ring motifs (constructed from N—H...O hydrogen bonds) along b axis, which are connected with discrete D₁¹(2) motifs (of C—H...O) along a

Table 2 Selected bond distances (Å) and angles (°).

P ₁ -O ₁	1.4770(9)	P ₁ -N ₁	1.6862(11)
P ₁ -N ₂	1.6335(11)	P ₁ -N ₃	1.6465(11)
C ₁ -O ₂	1.2254(15)		
O ₁ -P ₁ -N ₁	104.42(5)	O ₁ -P ₁ -N ₂	114.04(5)
O ₁ -P ₁ -N ₃	118.98(6)	N ₁ -P ₁ -N ₂	113.23(6)
N1-P1-N3	106.76(5)	N ₂ -P ₁ -N ₃	99.54(5)
P ₁ -N ₁ -C ₁	125.72(8)	P ₁ -N ₂ -C ₉	126.74(9)
P ₁ -N ₃ -C ₅	127.95(9)		

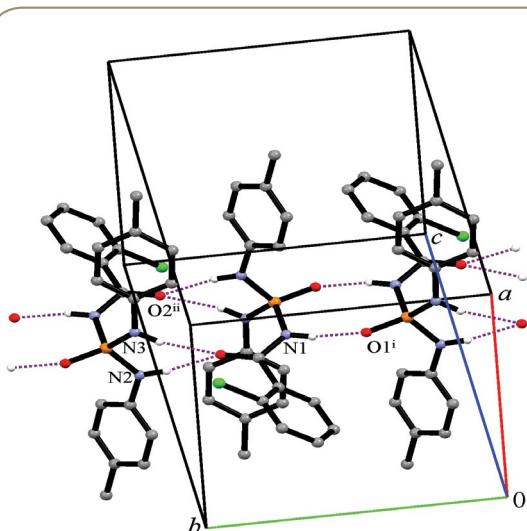


Figure 2 The crystal packing diagram for $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}] \text{P(O)}[\text{NHC}_6\text{H}_4\text{-4-CH}_3\text{]}_2$ is represented, showing a 1-D arrangement along the *b* axis. The H atoms bonded to C atoms were omitted for clarity. The $\text{N}_p\text{-H}\dots\text{O}=\text{C}$ and $\text{N}_{cp}\text{-H}\dots\text{O}=\text{P}$ hydrogen bonds are shown as dashed lines (symmetry codes: (i) $-x + 2, -y + 1, -z$; (ii) $-x + 2, -y + 2, -z$).

axis. Weak $\text{C-H}\dots\text{Cl}$ hydrogen bonds connect the slabs in the *c* axis direction, giving rise to a three-dimensional supramolecular structure.

Acknowledgements

Financial support of this work by Ferdowsi University of Mashhad is gratefully acknowledged (Project No. 28383/3). The X-ray part of the work was carried out with the support of Czech Science Foundation, grant GACR 15-12719S using instruments of the ASTRA lab established within the Operation program Prague Competitiveness - project CZ.2.16/3.1.00/24510.

Table 3. Hydrogen bonds geometries for $[2\text{-Cl-C}_6\text{H}_4\text{C(O)NH}] \text{P(O)}[\text{NHC}_6\text{H}_4\text{-4-CH}_3\text{]}_2$ (Å and °). Symmetry codes: (i) $-x + 2, -y + 1, -z$ (ii) $-x + 2, -y + 2, -z$ (iii) $x - 1, y, z$ (iv) $-x + 2, y - 1/2, -z + 1/2$.

<i>D-H</i> ... <i>A</i>	<i>d</i> (<i>D-H</i>)	<i>d</i> (<i>H</i> ... <i>A</i>)	<i>d</i> (<i>D</i> ... <i>A</i>)	∠ <i>DHA</i>
$\text{N}_1\text{-H}_1\text{n}_1\text{...O}_1^{\text{i}}$	0.874(13)	1.963(13)	2.8172(13)	165.6(15)
$\text{N}_2\text{-H}_1\text{n}_2\text{...O}_2^{\text{ii}}$	0.874(12)	2.005(12)	2.8552(14)	164.1(16)
$\text{N}_3\text{-H}_1\text{n}_3\text{...O}_2^{\text{ii}}$	0.874(14)	2.280(15)	3.0431(14)	145.8(15)
$\text{C}_{12}\text{-H}_1\text{c}_{12}\text{...O}_1^{\text{iii}}$	0.960	2.5388	3.319(2)	138.49
$\text{C}_{14}\text{-H}_1\text{c}_{14}\text{...Cl}_1^{\text{iv}}$	0.960	2.7843	3.353(2)	118.71

References

- Bollinger JC, Levy-Serpier J, Debord J, Penicaut B (1990) Acetylcholinesterase inhibition by two phosphoric 4-nitroanilides. *J Enzyme Inhib Med Chem* 3: 211-217.
- Andrews RK, Dexter A, Blakeley RL, Zerner B (1986) Jack Bean Urease (EC 3.5.1.5). 8. On the inhibition of urease by amides and esters of phosphoric acid. *J Am Chem Soc* 108: 7124-7125.
- Gholivand K, Ebrahimi Valmoozi AA, Mahzouni HR, Ghadimi S, Rahimi R, et al. (2013) Molecular docking and QSAR studies: noncovalent interaction between acephate analogous and the receptor site of human acetylcholinesterase. *J Agric Food Chem* 61: 6776-6785.
- Fu Z, Chivers T (2007) Solvent effects on the reactions of copper chlorides with $OP(NH-t-Bu)_3$ – Formation of the novel $[Cu_5Cl_{10}]^{5-}$ anion via in situ templation. *Can J Chem* 85: 358-365.
- Pourayoubi M, Golen JA, Rostami Chaijan M, Divjakovic V, Negari M, et al. (2011) The hydrogen-bonded dimers of N,N',N'' -tricyclohexylphosphoric triamide in new tin(IV) and copper(II) complexes. *Acta Crystallogr C67*: 160-164.
- Gholivand K, Mostaanazadeh H, Koval T, Dušek M, Erben MF, et al. (2010) Syntheses, spectroscopic study and X-ray crystallography of some new phosphoramidates and lanthanide(III) complexes of N -(4-nitrobenzoyl)- N' , N'' -bis(morpholino)phosphoric triamide. *Acta Crystallogr B* 66: 441-450.
- Gholivand K, Mahzouni HR, Pourayoubi M, Amiri S (2010) High-coordinated lanthanum(III) complexes with new mono- and bidentate phosphoryl donors; spectroscopic and structural aspects. *Inorg Chim Acta* 363: 2318-2324.
- Trush EA, Amirkhanov VM, Ochynniov VA, Swiatek-Kozłowska J, Lanikina KA, et al. (2003) Metal carbacylamidophosphates: ability of coordination patterns to di- and polymerization. *Polyhedron* 22: 1221-1229.
- Gholivand K, Pourayoubi M, Shariatnia Z (2007) $^{2,3}J(P,X)$ [$X = H, C$] coupling constants dependency on the ring size, hybridization, and substituents in new diazaphospholes and diazaphosphorinanes, NMR and X-ray crystallography studies. *Polyhedron* 26: 837-844.
- Pourayoubi M, Sabbaghi F (2009) Synthesis, spectroscopic characterization and crystal structure of a new acetyl phosphorylamidate $P(O)[NHC(O)C_6H_4(4-NO_2)][N(CH(CH_3)_2)(CH_2C_6H_5)]_2$. *J Chem Crystallogr* 39: 874-880.
- Pourayoubi M, Shoghpour S, Bruno G, Amiri Rudbari H (2011) N -(3-Fluorobenzoyl)- N',N'' -bis(4-methylphenyl)phosphoric triamide. *Acta Crystallogr E67*: 3034.
- Pourayoubi M, Shoghpour S, Bruno G, Amiri Rudbari H (2011) N,N' -Dicyclohexyl- N'' -(3-fluorobenzoyl)- N , N' -dimethylphosphoric triamide. *Acta Crystallogr E67*: 3028-3029.
- Rigaku Oxford Diffraction (2015) CrysAlisPRO, Rigaku Corporation, Tokyo, Japan.
- Palatinus L, Chapuis G (2007) SUPERFLIP – a computer program for the solution of crystal structures by charge flipping in arbitrary dimensions. *J Appl Crystallogr* 40: 786-790.
- Petříček V, Dušek M, Palatinus L (2014) Crystallographic computing system JANA2006: general features. *Z Kristallogr* 229: 345-352.
- Kirsanov AV, Makitra RG (1956) Dichlorides of acylamidophosphoric acids of aromatic series. *Zh Obshch Khim* 26: 905-907.
- Rudd MD, Lindeman SV, Husebye S (1996) Structural characteristics of three-coordinate arylhalide tellurium(II) complexes with chalcogen ligands. Synthesis, spectroscopic characterization and X-ray structural studies of bromo[N -methylbenzothiazole-2(3H)selone]phenyltellurium(II), bromophenyl[tris(dimethylamino)phosphaneselenide]-tellurium(II) and tris(dimethylamino) phosphanesulfide. *Acta Chem Scand* 50: 759-774.
- Corbridge DEC (1995) Phosphorus: An outline of its chemistry, biochemistry and technology. 5th edn. Amsterdam: Elsevier Science.
- Gilheany DG (1994) No d orbitals but Walsh diagrams and maybe banana bonds: chemical bonding in phosphines, phosphine oxides, and phosphonium Ylides. *Chem Rev* 94: 1339-1374.
- Pourayoubi M, Jasinski JP, Shoghpour Bayraq S, Eshghi H, Keeley AC, et al. (2012) Three new $[XC(O)NH]P(O)[N(CH_2-C_6H_5)_2]_2$ phosphoric triamides ($X = CClF_2$, $3-F-C_6H_4$ and $3,5-F_2-C_6H_3$): a database analysis of tertiary N-atom geometry in compounds with a $C(O)NHP(O)[N]_2$ core. *Acta Crystallogr C68*: 399-404.
- Pourayoubi M, Toghraee M, Divjakovic V, van der Lee A, Mancilla Percino T, et al. (2013) 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 Crystallogr B69*: 184-194.
- Pourayoubi M, Nečas M, Negari M (2012) The double H-atom acceptability of the $P=O$ group in new $XP(O)(NHCH_2C_6H_4-2-Cl)_2$ phosphoramidates [$X = C_6H_5O-$ and $CF_3C(O)NH-$]: a database analysis of compounds having a $P(O)(NHR)$ group. *Acta Crystallogr C68*: 51-56.
- Allen FH, Bruno IJ (2010) Bond lengths in organic and metal-organic compounds revisited: $X-H$ bond lengths from neutron diffraction data. *Acta Crystallogr B66*: 380-386.
- Pourayoubi M, Tarahhomi A, Rheingold AL, Golen JA (2010) N,N' -Dibenzyl- N'' -(2,6-difluorobenzoyl)- N,N' -dimethylphosphoric triamide. *Acta Crystallogr E66*: 2524.
- Pourayoubi M, Tarahhomi A, Rheingold AL, Golen JA (2010) N,N' -Di-tert-butyl- N'' -(2,6-difluorobenzoyl)phosphoric triamide. *Acta Crystallogr E66*: 3159.
- Pourayoubi M, Tarahhomi A, Rheingold AL, Golen JA (2011) N -(2-Fluorobenzoyl)- N',N'' -bis(4-methylphenyl)phosphoric triamide. *Acta Crystallogr E67*: 934.
- Pourayoubi M, Rostami Chaijan M, Torre-Fernández L, García-Granda S (2011) N -Benzoyl- N',N'' -dicyclohexylphosphoric triamide. *Acta Crystallogr E67*: 1360.
- Pourayoubi M, Rostami Chaijan M, Torre-Fernández L, García-Granda S (2011) N,N' -Dibenzyl- N,N' -dimethyl- N'' -(4-nitrobenzoyl) phosphoric triamide. *Acta Crystallogr E67*: 1031.
- Pourayoubi M, Saneei A (2011) N -(2-Chloro-2,2-difluoroacetyl)- N',N'' -diisopropylphosphoric triamide. *Acta Crystallogr E67*: 665.
- Pourayoubi M, Toghraee M, Divjakovic V (2011) N,N' -Bis(2-chlorobenzyl)- N'' -(dichloroacetyl)phosphoric triamide. *Acta Crystallogr E67*: 333.
- Raissi Shabari A, Pourayoubi M, Saneei A (2011) N,N' -Dibenzyl- N'' -(2-chloro-2,2-difluoroacetyl)- N,N' -dimethylphosphoric triamide. *Acta Crystallogr E67*: 663-664.
- Tarahhomi A, Pourayoubi M, Rheingold AL, Golen JA (2011) Different orientations of $C=O$ versus $P=O$ in $P(O)NHC(O)$ skeleton: the first study on an aliphatic diazaphosphorinane with a gauche orientation. *Struct Chem* 22: 201-210.
- Toghraee M, Pourayoubi M, Divjakovic V (2011) Study on H-bond patterns in phosphoric triamides having a $P(O)NHC(O)$ skeleton, a gauche orientation of $P(O)$ vs $C(O)$ in new compounds. *Polyhedron* 30: 1680-1690.