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X-Ray Single Crystal Structure Of 6-Phenyl-6-Selanylidene-13,13a Dihydro-6H, 8H-6λ^5-Quinazolino [3,2 c][1,3,2] Benzoxazaphosphinin-8-One

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Abstract

The title compound($C_{20}H_{15}N_2O_2PSe$) is synthesized and characterized by NMR, IR, GC-MS and single crystal X-ray diffraction. It has triclinic (P1̄) symmetry with unit cell parameters: a=8.1901 (5) Å, b= 10.6528 (5) Å, c= 11.8017 (8) Å, α=65.547 (1) °, β= 74.393 (2) °, γ = 80.434 (1) ° In this crystal structure, one P-atom attached with Se atom, one phenyl ring, one O-atom and one N-atom. The bond angles for Se—P—atom are 112.64 (6) °, 116.30 (7) °, 119.31(8) ° with O2, N1, C14 respectively. The P=Se bond length is 2.0735(7) Å. The other bond lengths are 1.612(1) Å, 1.685(2) Å, 1.793(2) Å for P1—O2, P1—N1, P1—C14 respectively.

Keywords: Single Crystal; Benzoxazaphosphinine; Selanylidene; Antibacterial

Introduction

Organophosphorus heterocycles like oxazaphosphinines with O and N in six-membered rings exhibit strong antitumor [1] and antibacterial activity [2]. As a result, there is a great deal of interest in drug design and synthesis [3] due to the therapeutic importance and stereochemical properties of oxazaphosphinines. The single crystal structure of the title compound is being reported as part of our research in this area ($C_{20}H_{15}N_2O_2PSe$).

Material and Methods

The synthesis, crystallisation, and spectroscopic studies of novel 6-phenyl-6-selanylidene 13,13a-dihydro-6H,8H-6λ^5-quinazolino[3,2-c][1,3,2]benzoxazaphosphinin-8-one has been reported previously [4]. In a reaction mixture of 6-Phenyl-12,12a-dihydro-5-oxa-6a,12-diaza-6-phospha-benzo[a]anthracen-7-one in toluene , an access of selenium powder (0.77 g, 10 mmol) was added and refluxed for 5 h. The resulting dark red colour solution was filtered hot under inert atmosphere and concentrated under reduced pressure. The crude product was crystallized with diethyl ether. (Melting point- 160-170° C); ^{31}P-NMR(161.5 MHz, $CDCl_3$): δ 73.4 (t) ($^1J_{P=Se}$: 895.66Hz); ^{13}C-NMR (100 MHz, $CDCl_3$): δ 165, 148, 146, 137, 135, 133, 132, 131, 129, 128, 125, 121, 117, 67, 45; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 6-8(m,13H), δ 4(broad, NH); IR Data: 578.64(P=Se), 1670.35 (C=O Stretching), 3282.84 (2° NH Stretching); MS (EI, 30 eV)m/z= 424 (62%, M+), , 267 (100%, base peak).

Single Crystal X-Ray Diffraction

A suitable single crystal of title compound for data collection was selected and data were collected at 273K by ω scan technique on three circle Bruker SMART diffractometer with CCD area detector using graphite monochromate radiation MoKα (λ=0.71073Å) from sealed X-ray tube diffraction source. The data collection was performed by using the Bruker APEX2 [5]. The data reduction and cell refinement were performed using the Bruker SAINT [6]. Molecular graphics was performed by using Olex2 1.5 [7]. The figures were produced using ORTEP [8] and PLATON [9]. Table 1 contains an overview of the crystal data, experimental details, and refining results.

Table 1: Experimental details.

Crystal Data	
Chemical formula	$C_{20}H_{15}N_2O_2PSe$
Mr	425.27
Crystal system, space group	Triclinic, P$\bar{1}$
Temperature (K)	273
a, b, c (Å)	8.1901 (5), 10.6528 (5), 11.8017 (8)
α, β, γ (°)	65.547 (1), 74.393 (2), 80.434 (1)
V (Å^3)	900.98 (9)
Z	2
Radiation type	Mo Kα
μ (mm^{-1})	2.19
Crystal size (mm)	0.24 × 0.17 × 0.11
Data Collection	
Diffractometer	Bruker SMART CCD 6000 area detector
Absorption correction	Multi-scan
No. of measured, independent and observed [I > 2σ(I)] reflections	10439, 2973, 2801
R_{int}	0.022
(Sin θ/λ)max (Å-1)	0.582
Refinement	
R[F^2 > 2σ(F^2)], wR(F^2), S	0.025, 0.066, 1.06
No. of reflections	2890
No. of parameters	235
H-atom treatment	H-atom parameters constrained
Δρmax, Δρmin (e Å^{-3})	0.43, -0.43

Geometric Parameters

The bond lengths are given in table 2, bond angles are given in table 3. And torsion angles are given in table 4.

Table 2: Bond lengths(Å).

Se1—P1	2.0735(7)	C15—C16	1.381(4)
P1—O2	1.612(1)	C6—H6	0.93
P1—N1	1.685(2)	C6—C5	1.370(4)
P1—C14	1.793(2)	C13—H13	0.93
O2—C20	1.402(2)	C13—C12	1.380(4)
O1—C1	1.213(3)	C10—H10	0.93
N1—C8	1.478(2)	C10—C11	1.378(5)
N1—C1	1.394(3)	C19—H19	0.93
N2—C8	1.447(3)	C19—C18	1.381(4)
N2—C7	1.388(3)	C3—H3	0.93
C8—H8	0.98	C3—C4	1.372(5)
C8—C9	1.505(3)	C11—H11	0.93

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C7—C2	1.402(3)	C11—C12	1.381(4)
C7—C6	1.400(4)	C5—H5	0.93
C14—C15	1.385(3)	C5—C4	1.391(3)
C14—C19	1.378(4)	C16—H16	0.93
C2—C1	1.472(4)	C16—C17	1.365(6)
C2—C3	1.393(4)	C12—H12	0.93
C20—C9	1.382(3)	C18—H18	0.93
C20—C13	1.377(3)	C18—C17	1.377(7)
C9—C10	1.390(3)	C4—H4	0.93
C15—H15	0.93	C17—H17	0.93
N2—H2	0.86		

Table 3: Bond angles(°).

Se1—P1—O2	112.64(6)	C14—C15—C16	118.7(2)
Se1—P1—N1	116.30(7)	H15—C15—C16	120.7
Se1—P1—C14	119.31(8)	C7—C6—H6	120.2
O2—P1—N1	99.50(9)	C7—C6—C5	119.7(2)
O2—P1—C14	99.9(1)	H6—C6—C5	120.1
N1—P1—C14	106.3(1)	C20—C13—H13	120.7
P1—O2—C20	118.3(1)	C20—C13—C12	118.6(2)
P1—N1—C8	123.1(1)	H13—C13—C12	120.7
P1—N1—C1	119.7(2)	C9—C10—H10	119.7
C8—N1—C1	116.9(2)	C9—C10—C11	120.6(2)
H2—N2—C8	123.1	H10—C10—C11	119.7
H2—N2—C7	123.2	C14—C19—H19	120.4
C8—N2—C7	113.7(2)	C14—C19—C18	119.3(3)
N1—C8—N2	106.6(2)	H19—C19—C18	120.3
N1—C8—H8	108.1	C2—C3—H3	119.8
N1—C8—C9	113.4(2)	C2—C3—C4	120.4(3)
N2—C8—H8	108.2	H3—C3—C4	119.8
N2—C8—C9	112.1(2)	C10—C11—H11	119.9
H8—C8—C9	108.2	C10—C11—C12	120.4(3)
N2—C7—C2	118.9(2)	H11—C11—C12	119.7
N2—C7—C6	121.9(2)	C6—C5—H5	119.4
C2—C7—C6	119.2(2)	C6—C5—C4	12132(3)
P1—C14—C15	121.0(2)	H5—C5—C4	119.4
P1—C14—C19	117.9(2)	C15—C16—H16	119.8
C15—C14—C19	121.1(2)	C15—C16—C17	120.4(3)
C7—C2—C1	120.2(2)	H16—C16—C17	119.8
C7—C2—C3	119.9(2)	C13—C12—C11	120.1(3)
C1—C2—C3	119.3(2)	C13—C12—H12	119.9
O2—C20—C9	120.1(2)	C11—C12—H12	120
O2—C20—C13	117.2(2)	C19—C18—H18	120.2

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C9—C20—C13	122.7(2)	C19—C18—C17	119.7(3)
C8—C9—C20	123.4(2)	H18—C18—C17	120.1
C8—C9—C10	119.1(2)	C3—C4—C5	119.5(3)
C20—C9—C10	117.6(2)	C3—C4—H4	120.2
O1—C1—N1	120.4(2)	C5—C4—H4	120.3
O1—C1—C2	125.1(2)	C16—C17—C18	120.8(4)
N1—C1—C2	114.3(2)	C16—C17—H17	119.6
C14—C15—H15	120.6	C18—C17—H17	119.6

Result And Discussion

Structure Description

The structure of the title compound has triclinic (P1̄) symmetry, in which one P-atom attached with Se-atom, one phenyl ring, one O-atom and one N-atom. The structure has 5 six member rings, in which 3 rings are almost coplanner and the two saturated rings which have heteroatoms are out of the plane due to rigidity (Figure 1-3). The bond angles for Se—P—atom are 112.64 (6)°, 116.30 (7)°, 119.31(8)° with O2, N1, C14 respectively. The P=Se bond length is 2.0735(7) Å the other bond lengths are 1.612(1), 1.685(2), 1.793(2) Å for P1—O2, P1—N1, P1—C14 respectively. The dihedral angle between the planes produce through the bonded atoms of two aromatic rings (C14---C19 and C2---C7) is 71.73°, dihedral angle between the planes produce through the bonded atoms of the aromatic ring(C14---C19) and heterocyclic ring (N1-C8-C20-C9-P1-O2) is 86.26°, and dihedral angle between the planes produce through the bonded atoms of the aromatic ring (C2---C7) and heterocyclic ring (N1-C8-C20-C9-P1-O2) atoms is 38.34° (Figure 4).

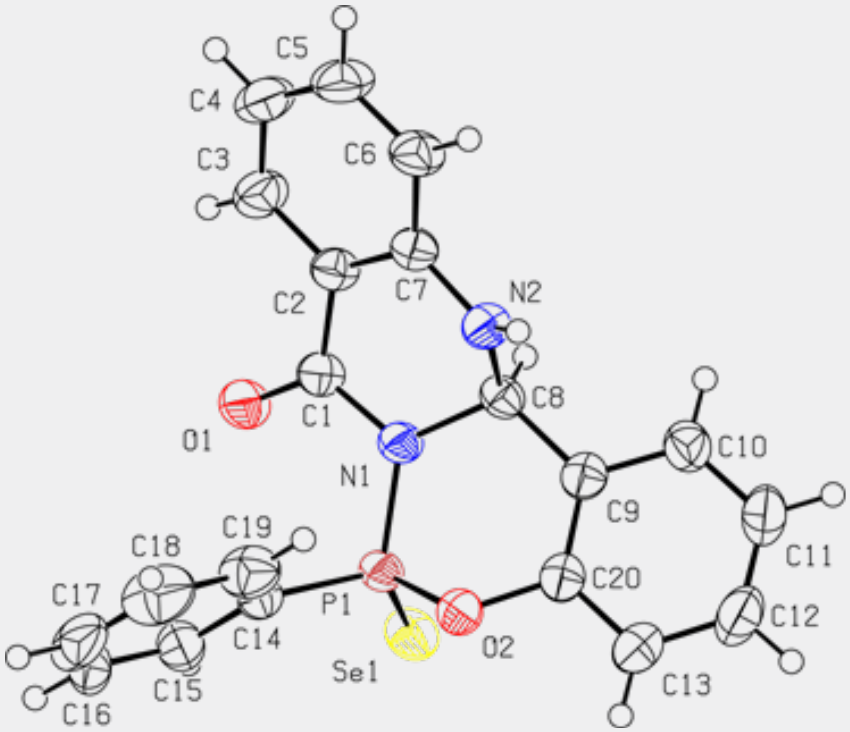

Figure 1: Ellipsoid plot of the title compound.

Supramolecular Feature

The intermolecular hydrogen-bonding is observed in this molecule, which may help to stabilize the crystal structure of molecule (Figure 5).In the crystal, the molecules are held together by an intermolecular interactions of the types C15—H15---N2 hydrogen-bond between the phenyl C—H and the pyrimidine

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nitrogen atom, C18—H18---O2 hydrogen bond between the phenyl C—H and the phosphorus heterocyclic ring oxygen atom and C6—H6---O1 hydrogen-bond between the phenyl C—H and the carbonyl oxygen atom (Table 5).

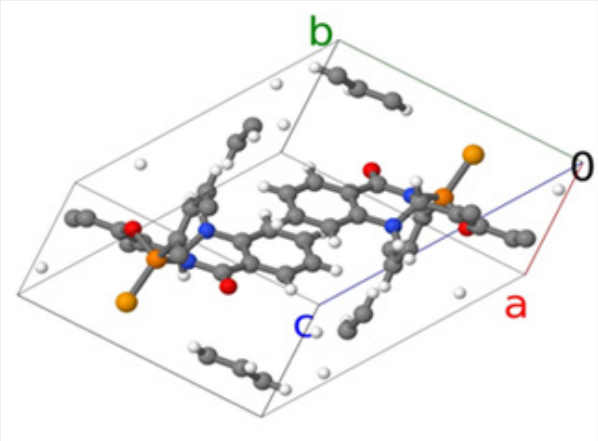

Figure 2: Molecular packing arrangement in unit cell.

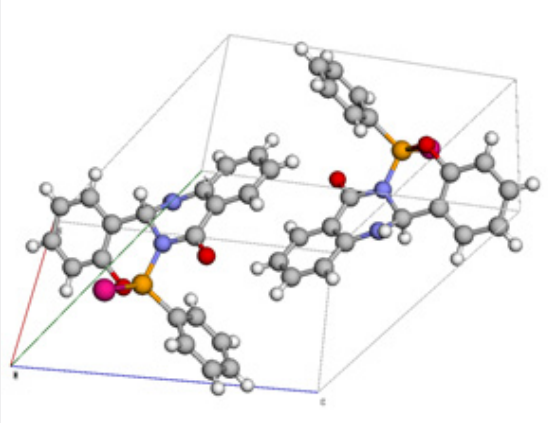

Figure 3: Molecular structure of title compound.

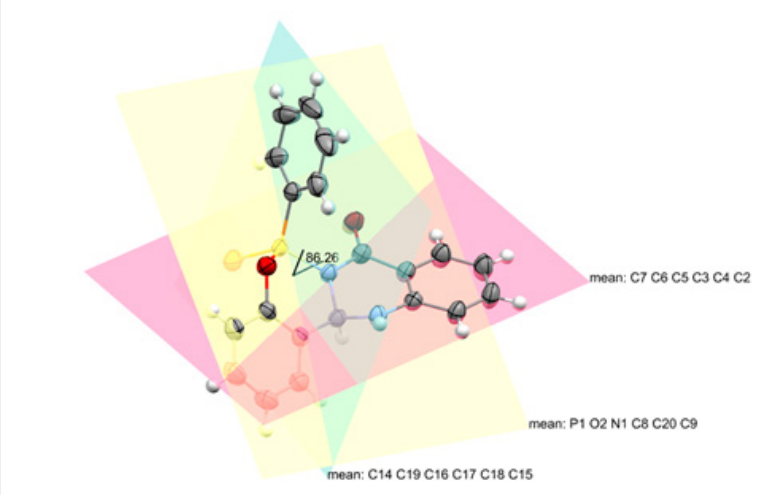

Figure 4: 3 planes showing in crystal with dihedral angles.

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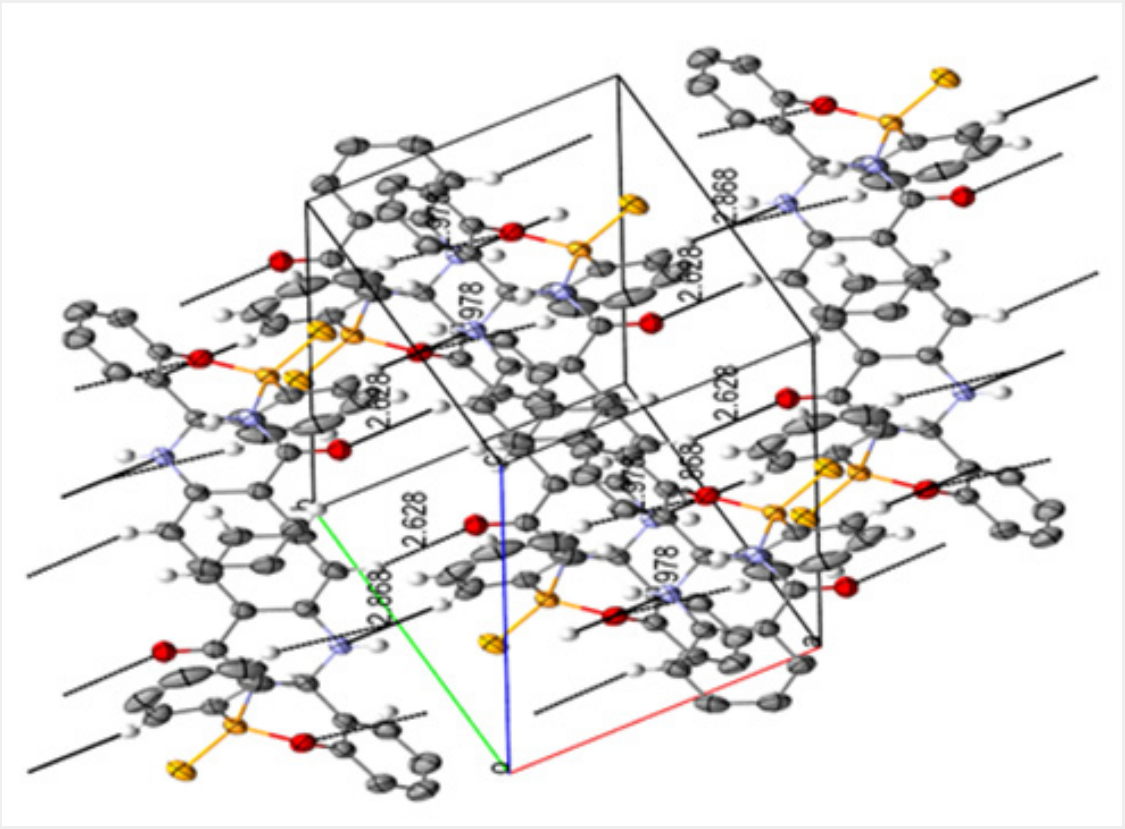

Figure 5: The crystal packing of the title compound. The H-bonds are shown as dashed lines. H-atoms are not involved in hydrogen bonding have been omitted for clarity.

Table 4: Torsion angles(°).

Se1—P1—O2—C20	71.2(2)	C7—C2—C1—N1	-9.4(3)
N1—P1—O2—C20	-52.6(2)	C3—C2—C1—O1	-5.8(4)
C14—P1—O2—C20	-161.1(2)	C3—C2—C1—N1	179.2(2)
Se1—P1—N1—C8	-85.3(2)	C7—C2—C3—H3	179.9
Se1—P1—N1—C1	87.8(2)	C7—C2—C3—C4	-0.2(4)
O2—P1—N1—C8	35.9(2)	C1—C2—C3—H3	-8.7
O2—P1—N1—C1	-151.0(2)	C1—C2—C3—C4	171.2(2)
C14—P1—N1—C8	139.2(2)	O2—C20—C9—C8	0.8(3)
C14—P1—N1—C1	-47.7(2)	O2—C20—C9—C10	179.7(2)
Se1—P1—C14—C15	-10.6(2)	C13—C20—C9—C8	-176.9(2)
Se1—P1—C14—C19	167.9(2)	C13—C20—C9—C10	2.0(4)
O2—P1—C14—C15	-133.7(2)	O2—C20—C13—H13	2.1
O2—P1—C14—C19	44.8(2)	O2—C20—C13—C12	-177.9(2)
N1—P1—C14—C15	123.3(2)	C9—C20—C13—H13	179.9
N1—P1—C14—C19	-58.2(2)	C9—C20—C13—C12	-0.1(4)

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P1—O2—C20—C9	39.9(3)	C8—C9—C10—H10	-3.1
P1—O2—C20—C13	-142.3(2)	C8—C9—C10—C11	176.8(2)
P1—N1—C8—N2	-129.9(2)	C20—C9—C10—H10	177.9
P1—N1—C8—H8	114	C20—C9—C10—C11	-2.1(4)
P1—N1—C8—C9	-6.0(2)	C14—C15—C16—H16	179
C1—N1—C8—N2	56.9(2)	C14—C15—C16—C17	-1.0(4)
C1—N1—C8—H8	-59.2	H15—C15—C16—H16	-1
C1—N1—C8—C9	-179.2(2)	H15—C15—C16—C17	179.1
P1—N1—C1—O1	-12.5(3)	C7—C6—C5—H5	179.7
P1—N1—C1—C2	162.7(2)	C7—C6—C5—C4	-0.3(4)
C8—N1—C1—O1	161.0(2)	H6—C6—C5—H5	-0.3
C8—N1—C1—C2	-23.8(3)	H6—C6—C5—C4	179.7
H2—N2—C8—N1	121.9	C20—C13—C12—C11	-1.7(4)
H2—N2—C8—H8	-122	C20—C13—C12—H12	178.2
H2—N2—C8—C9	-2.8	H13—C13—C12—C11	178.3
C7—N2—C8—N1	-58.1(2)	H13—C13—C12—H12	-1.8
C7—N2—C8—H8	58	C9—C10—C11—H11	-179.7
C7—N2—C8—C9	177.2(2)	C9—C10—C11—C12	0.4(4)
H2—N2—C7—C2	-151.4	H10—C10—C11—H11	0.3
H2—N2—C7—C6	26	H10—C10—C11—C12	-179.6
C8—N2—C7—C2	28.6(3)	C14—C19—C18—H18	179.1
C8—N2—C7—C6	-154.0(2)	C14—C19—C18—C17	-0.9(5)
N1—C8—C9—C20	-16.9(3)	H19—C19—C18—H18	-0.9
N1—C8—C9—C10	164.2(2)	H19—C19—C18—C17	179.1
N2—C8—C9—C20	104.0(2)	C2—C3—C4—C5	-0.8(4)
N2—C8—C9—C10	-75.0(3)	C2—C3—C4—H4	179.1
H8—C8—C9—C20	-136.9	H3—C3—C4—C5	179.1
H8—C8—C9—C10	44.2	H3—C3—C4—H4	-1
N2—C7—C2—C1	7.2(3)	C10—C11—C12—C13	1.6(5)
N2—C7—C2—C3	178.4(2)	C10—C11—C12—H12	-178.3
C6—C7—C2—C1	-170.3(2)	H11—C11—C12—C13	-178.3
C6—C7—C2—C3	1.0(3)	H11—C11—C12—H12	1.8
N2—C7—C6—H6	1.9	C6—C5—C4—C3	1.1(5)
N2—C7—C6—C5	-178.1(2)	C6—C5—C4—H4	-178.8
C2—C7—C6—H6	179.3	H5—C5—C4—C3	-178.9
C2—C7—C6—C5	-0.7(4)	H5—C5—C4—H4	1.2
P1—C14—C15—H15	-1.2	C15—C16—C17—C18	0.6(5)
P1—C14—C15—C16	178.8(2)	C15—C16—C17—H17	-179.3
C19—C14—C15—H15	-179.7	H16—C16—C17—C18	-179.3
C19—C14—C15—C16	0.4(4)	H16—C16—C17—H17	0.7
P1—C14—C19—H19	2.1	C19—C18—C17—C16	0.3(6)
P1—C14—C19—C18	-177.9(2)	C19—C18—C17—H17	-179.7

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C15—C14—C19—H19	-179.4	H18—C18—C17—C16	-179.7
C15—C14—C19—C18	0.6(4)	H18—C18—C17—H17	0.3
C7—C2—C1—O1	165.5(2)		

Table 5: Hydrogen-bond geometry (Å, °).

D—H---A	D—H	H---A	D---A	D—H---A
C15—H15---N2	0.93	2.868	3.790(5)	171
C18—H18---O2	0.93	2.978	3.832(5)	153.3
C6—H6---O1	0.93	2.628	3.472(3)	151.3

Supplementary Information

CCDC 2192461 contains crystallographic supplementary data for this paper.

Conclusion

The structure of the title compound has triclinic (P1̄) symmetry. The bond angles for Se—P—atom are 112.64 (6)°, 116.30 (7)°, 119.31(8) °with O2, N1, C14 respectively. The P=Se bond length is 2.0735(7) Å. The other bond lengths are 1.612(1), 1.685(2), 1.793(2) Å for P1—O2, P1—N1, P1—C14 respectively. The dihedral angle between the planes produce through the bonded atoms of two aromatic rings (C14---C19 and C2---C7) is 71.73°, dihedral angle between the planes produce through the bonded atoms of the aromatic ring(C14---C19) and heterocyclic ring (N1-C8-C20-C9-P1-O2) is 86.26°, and dihedral angle between the planes produce through the bonded atoms of the aromatic ring (C2---C7) and heterocyclic ring (N1-C8-C20-C9-P1-O2) atoms is 38.34°.

The intermolecular hydrogen-bonding is observed in this molecule, which may help to stabilize the crystal structure of molecule. In the crystal, the molecules are held together by an intermolecular interactions of the types C15-H15---N2 hydrogen-bond between the phenyl C-H and the pyrimidine nitrogen atom, C18-H18---O2 hydrogen bond between the phenyl C-H and the phosphorus heterocyclic ring oxygen atom and C6-H6---O1 hydrogen-bond between the phenyl C-H and the carbonyl oxygen atom.

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