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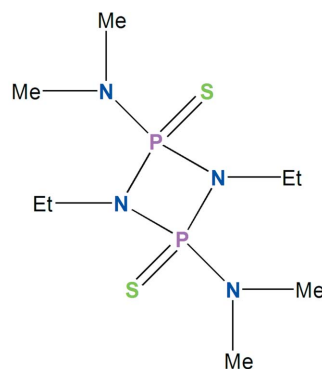
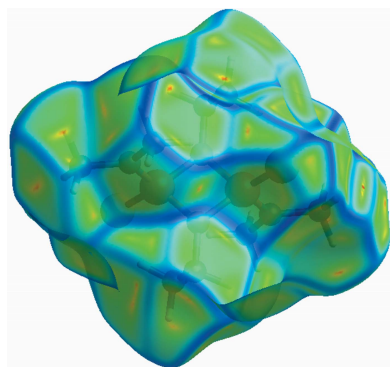
Crystal structure and Hirshfeld surface analysis of the new cyclodiphosphazane [EtNP(S)NMe₂]₂

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The cyclic compound 2,4-bis(dimethylamino)-1,3-diethylcyclodiphosphazane-2,4-dithione [systematic name: 2,4-bis(dimethylamino)-1,3-diethyl-1,3,2λ⁵,4λ⁵-diazadiphosphetidine-2,4-dithione], C₈H₂₂N₄P₂S₂ or [EtNP(S)NMe₂]₂, is member of a class of molecules that may be used, by virtue of their complexation properties, for the extraction of metals. This compound was characterized in solution by (¹H and ³¹P) NMR, and in the solid state by energy-dispersive X-ray spectroscopy (EDX) and by X-ray crystallography. In the crystal, the molecule sits on an inversion centre such that the P and N atoms form a centrosymmetric cyclic P₂N₂ arrangement. The crystal packing is dominated by van der Waals interactions. The prevalence of these interactions is illustrated by an analysis of the three-dimensional Hirshfeld surface (HS) and by two-dimensional fingerprint plots (FP). The relative contribution of different interactions to the HS indicates that the H···H contacts account for 74.3% of the total HS area.

1. Chemical context

In the study of organophosphorus compounds, one of the aims is to prepare new complexing agents. Indeed, the literature shows many studies of the bidentate organophosphorus ligands HN[P(E)R₂]₂ (E: O, S, Se; Balazs *et al.* 1999; Silvestru *et al.* 2000; Ghesner *et al.* 2005; Cristurean *et al.* 2008) and RN[P(E)R₂]₂ (Benabicha *et al.* 1986; Ladeveze *et al.* 1986; Alouani *et al.* 2002, 2007; Peulecke *et al.* 2009), *etc.* All of these ligands may act as chelating agents containing both hard (N) and soft (P) elements. In addition, the flexibility of the (EPNPE) system provides a ready means of altering, and thereby possibly improving, their complexing properties. Several complexes based on these ligands have been reported, such as those described by Bennis & Alouani (2012) and by Mejri *et al.* (2016).



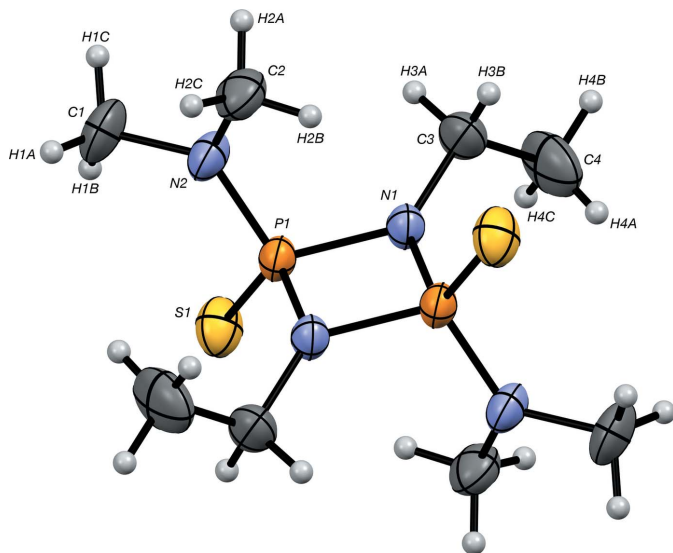


Figure 1
The molecular structure of (I). Atomic displacement parameters for the non-H atoms are drawn at the 30% probability level. Unlabelled atoms are related to labelled ones by the symmetry operation $-x + 1, -y + 1, -z$.

We report here the synthesis, characterization by (^1H and ^{31}P) NMR and energy-dispersive X-ray (EDX) spectroscopies, and a single-crystal structure of a new cyclodiphosphazane, 1,3-diethyl-2,4-dimethylamine-2,4-dithiocyclodiphosphazane, $[\text{EtNP}(\text{S})\text{NMe}_2]_2$ (I). In order to evaluate the nature of the intermolecular interactions in the crystal packing and their associated energies, detailed analyses of Hirshfeld surfaces (HS) and fingerprint plot (FP) calculations were performed (Spackman & McKinnon, 2002; Parkin *et al.*, 2007; Rohl *et al.*, 2008; Spackman & Jayatilaka, 2009).

2. Structural commentary

The molecular structure of (I) is shown in Fig. 1, selected crystallographic data are presented in Table 1, and an EDX

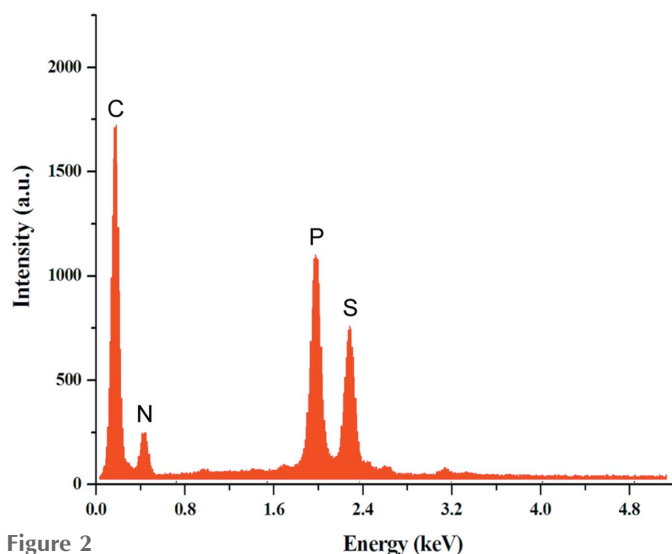


Figure 2
The EDX spectrum of (I), showing the presence of C, N, P, and S.

spectrum confirming the presence of C, N, P and S is shown in Fig. 2.

Each phosphorus atom is bonded to one sulfur and three nitrogen atoms, which are linked to methyl or ethyl groups. Atoms P1 and N1 form a centrosymmetric cyclic P_2N_2 arrangement about an inversion center $(\frac{1}{2}, \frac{1}{2}, 0)$. The P1–N1 distances in the ring [1.6856 (17) and 1.6719 (16) Å] are longer than the P1–N2 distance [1.6325 (19) Å], and the P1–S1 distance is 1.9291 (9) Å. These geometric parameters are in agreement with those observed in related non-cyclic and cyclic neutral ligands (Hill *et al.*, 1994; Alouani *et al.*, 2002; Peulecke *et al.*, 2009; Chandrasekaran *et al.* 2011).

With regard to the conformation of (I), its structure differs from that of $\text{P}_2\text{S}_2\text{N}_5\text{C}_9\text{H}_{27}$ (S-NIPA) (Benabicha *et al.* 1986) primarily by the existence of the P_2N_2 ring. The literature also shows several similar ligands, for example *trans*- $[(\text{EtNH})\text{P}(\text{S})\text{N}(\text{Et})_2]_2$ (Hill *et al.* 1994) and *cis*- $\text{P}_2\text{S}_2\text{N}_4\text{C}_{20}\text{H}_{42}$ (Chandrasekaran *et al.* 2011). The most similar known ligand to (I) is the cyclic molecule *trans*- $[(\text{EtNH})\text{P}(\text{S})\text{N}(\text{Et})_2]_2$ (Hill *et al.* 1994). The two molecules differ in the environments of the nitrogen atoms, which are all bound to ethyl groups in *trans*- $[(\text{EtNH})\text{P}(\text{S})\text{N}(\text{Et})_2]_2$, the peripheral carbons of which are all disordered.

3. Supramolecular features

A perspective view of (I) is presented in Fig. 3. Although there are several intra- and intermolecular close contacts of the form $\text{C}-\text{H}\cdots\text{A}$ ($\text{A} = \text{S}, \text{N}$), no classical hydrogen bonds are found and the dominant interactions are van der Waals contacts.

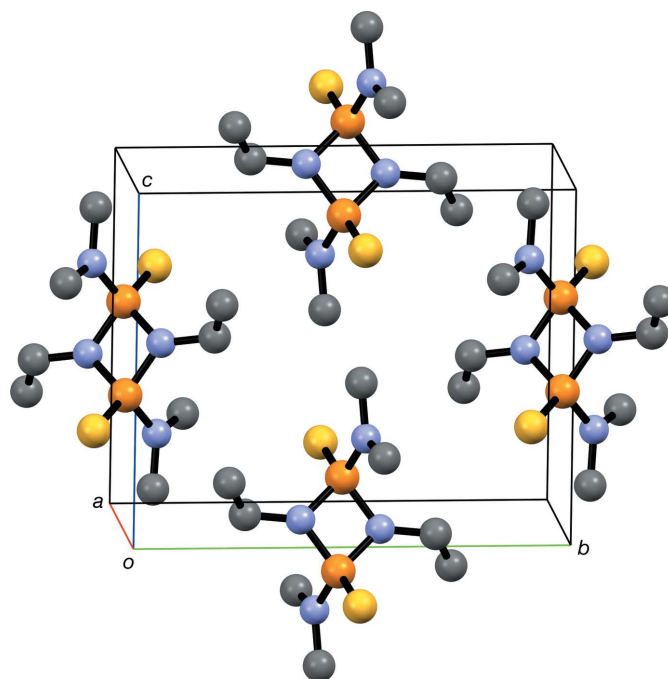


Figure 3
Perspective view of part of the crystal structure of (I), viewed approximately down the *a* axis. H atoms have been omitted for clarity.

4. Hirshfeld surface analysis

Organic small molecule crystal packings are often dominated by a particular type of interaction, e.g. hydrogen bonding or van der Waals contacts. However, the overall crystal packing is determined by a combination of many forces, and hence all of the intermolecular interactions of a structure should be taken into account. Visualization and exploration of intermolecular close contacts of a structure is invaluable, and this can be achieved using the Hirshfeld surface (Spackman & McKinnon, 2002; Spackman & Jayatilaka, 2009). A large range of properties can be visualized on the Hirshfeld surface with the program *CrystalExplorer* (Wolff *et al.*, 2012), including d_e and d_i , which represent the distances from a point on the HS to the nearest atoms outside (external) and inside (internal) the surface, respectively.

Intermolecular distance information on the surface can be condensed into a two-dimensional histogram of d_e and d_i , which is a unique identifier for molecules in a crystal structure, and is known as a fingerprint plot (Parkin *et al.*, 2007; Rohl *et al.*, 2008). Instead of plotting d_e and d_i on the Hirshfeld surface, contact distances are normalized in *CrystalExplorer* using the van der Waals radius of the appropriate internal (r_i^{vdw}) and external (r_e^{vdw}) atom of the surface:

$$d_{\text{norm}} = (d_i - r_i^{\text{vdw}})/r_i^{\text{vdw}} + (d_e - r_e^{\text{vdw}})/r_e^{\text{vdw}}$$

For (I), the three-dimensional HS mapped over d_{norm} is given in Fig. 4. Contacts with distances equal to the sum of the van der Waals radii are shown in white, and contacts with distances shorter than or longer than the related sum values are shown in red (highlighted contacts) or blue, respectively. Two-dimensional FP plots showing the occurrence of all kinds of intermolecular contacts are presented in Fig. 5a.

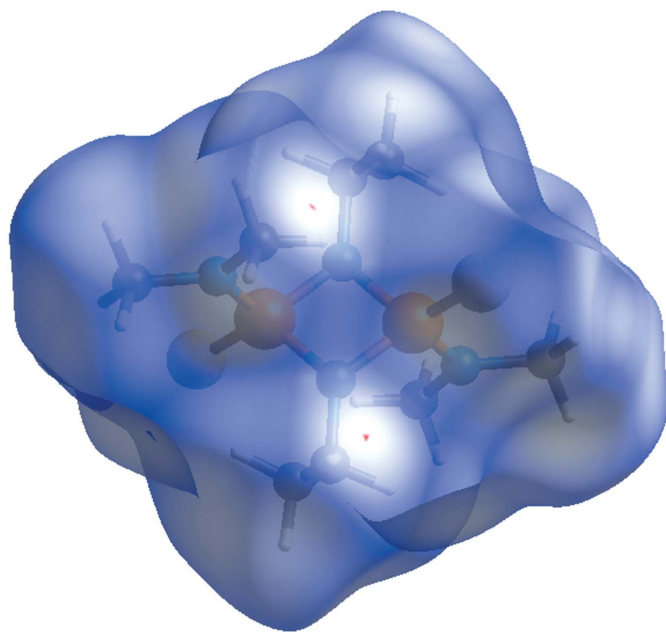


Figure 4
View of the three-dimensional Hirshfeld surface (HS) of (I) mapped with d_{norm} .

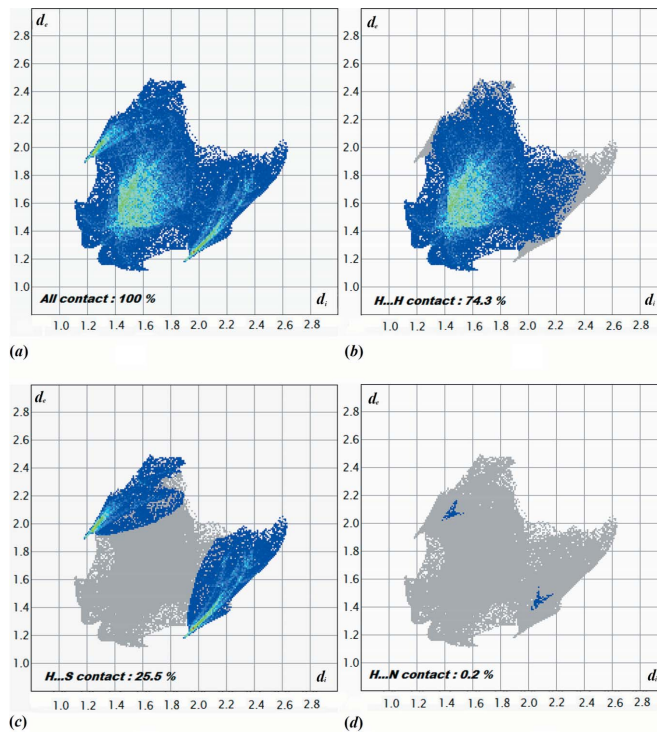


Figure 5

The two-dimensional fingerprint plots of (I), showing (a) all interactions, and delineated into (b) H...H, (c) H...S and (d) H...N interactions [d_e and d_i represent the distances from a point on the HS to the nearest atoms outside (external) and inside (internal) the surface, respectively].

The H...H interactions are shown on the three-dimensional HS as white spots. These contacts appear in the middle of the scattered points in the two-dimensional FP (Fig. 5b), and represent the most significant contribution to the overall

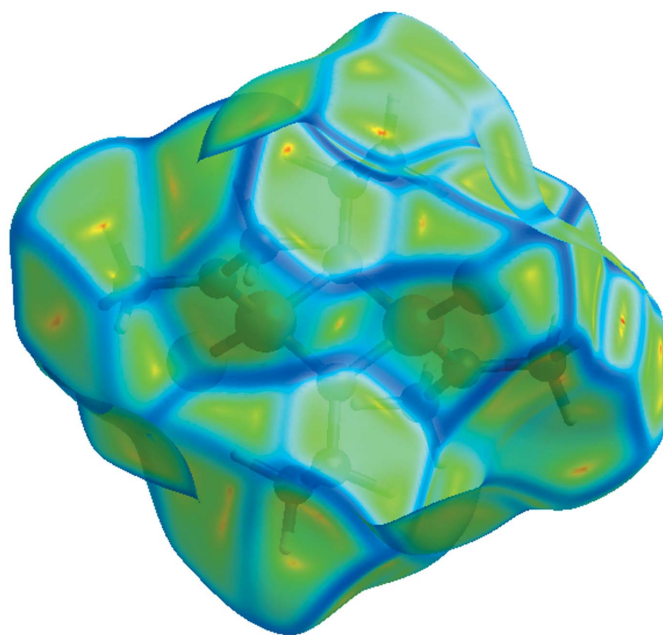


Figure 6
Hirshfeld surface of (I) mapped over curvedness.

Table 1
Experimental details.

Crystal data	
Chemical formula	C ₈ H ₂₂ N ₄ P ₂ S ₂
<i>M_r</i>	300.35
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	7.1975 (10), 11.448 (2), 9.645 (2)
β (°)	96.39 (3)
<i>V</i> (Å ³)	789.8 (2)
<i>Z</i>	2
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.52
Crystal size (mm)	0.40 × 0.40 × 0.30
Data collection	
Diffractometer	Enraf–Nonius CAD-4
Absorption correction	ψ scan (North <i>et al.</i> , 1968)
<i>T_{min}</i> , <i>T_{max}</i>	0.999, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	3150, 1724, 1463
<i>R_{int}</i>	0.016
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.638
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.041, 0.127, 1.08
No. of reflections	1724
No. of parameters	76
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.28, -0.27

Computer programs: *CAD-4 EXPRESS* (Duisenberg, 1992; Maciček & Yordanov, 1992), *XCAD4* (Harms & Wocadlo, 1995), *SHELXS97* (Sheldrick, 2008), *SHELXL2014/7* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2006), *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

three-dimensional HS (74.3%). Significant H···S/S···H interactions (25.5%) can also be seen, indicated by the pair of wings in the two-dimensional FP with a prominent long spike at *d_e* + *d_i* ~ 1.9 Å (Fig. 5c). The H···N/N···H interactions are shown on the three-dimensional HS marked with a blue spot for long contacts. These comprise only 0.2% of the total Hirshfeld surface, and are represented by two symmetrical narrow pointed spikes with *d_e* + *d_i* ~ 2 Å (Fig. 5d). The presence of these interactions may also be shown by the Hirshfeld surface mapped as a function of curvedness (Fig. 6).

5. Synthesis and crystallization

All reagents and solvents were obtained from commercial sources and used without further purification. The synthesis of (I) was carried out in three steps:

- Step 1: Addition of pyridine dropwise to a solution in anhydrous heptane of 2 mol of (EtNH₂HCl) and 2 mol of PCl₃ at 268 K, gave precipitation in the form of a salt. Then, the reaction mixture was refluxed for 24 h. An oil was obtained after filtration of the pyridinium salt and evaporation of the heptane and the excess PCl₃. This step corresponds to the formation of P₂N₂ cycle, according to the bibliographic data (Chandrasekaran *et al.* 2011; Hill *et al.* 1994). All these operations were conducted under a nitrogen atmosphere to avoid hydrolysis of the chlorinated compounds. The yield of this step is 85% with respect to ethylammonium chloride.

- Step 2: At a temperature of 263 K, 1 mol of the synthesized [EtNPCI]₂ was added dropwise to an ether solution containing 2 mol of dimethylamine, 2 mol of triethylamine and 4-dimethylaminopyridine (4-DMAP) as catalyst. After 10 h of agitation, Et₃NHCl was precipitated. Filtration of the salt and evaporation of the ether gave an oil. All these operations were conducted under a nitrogen atmosphere. The yield of this step is 40%.

- Step 3: The sulfurization of [EtNPNMe₂]₂ with 2 mol of sulfur gave the final product, 1,3-diethyl-2,4-dimethyl-2,4-dithio-cyclodiphosphazane (I), in a yield of about 80%.

Crystallization was carried out from ethanol by slow evaporation at room temperature. After one week, yellow single crystals suitable for X-ray diffraction analysis were obtained. A qualitative EDX analysis on some crystals confirmed the presence of C, N, P and S.

Yield: (80%), yellow solid, ¹H NMR (300 MHz, CDCl₃): δ (ppm) 1.17 (*t*, 1H, ³*J*_{HH} = 7.26 Hz), 2.91 (*d*, 1H, ³*J*_{HP} = 12.45 Hz), 3.03 (*m*, 2H); ³¹P NMR (300 MHz, CDCl₃): δ (ppm) 60.13 (1P).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms attached to CH₃ and CH₂ groups were placed geometrically and refined using a riding model: C–H = 0.96 Å for CH₃ group with *U*_{iso}(H) = 1.5*U*_{eq}(C) and C–H = 0.97 Å for CH₂ group with *U*_{iso}(H) = 1.2*U*_{eq}(C).

Acknowledgements

Retired Professor Ahmed Driss, University of Tunis El Manar, Faculty of Sciences of Tunis, is thanked for his assistance in the measurement of the X-ray data.

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Computing details

Data collection: *CAD-4 EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992); cell refinement: *CAD-4 EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992); data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014/7* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

2,4-Bis(dimethylamino)-1,3-diethyl-1,3,2λ⁵,4λ⁵-diazadiphosphetidine-2,4-dithione

Crystal data

C₈H₂₂N₄P₂S₂

M_r = 300.35

Monoclinic, *P*2₁/*c*

a = 7.1975 (10) Å

b = 11.448 (2) Å

c = 9.645 (2) Å

β = 96.39 (3)°

V = 789.8 (2) Å³

Z = 2

F(000) = 320

D_x = 1.263 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 10–15°

μ = 0.52 mm⁻¹

T = 293 K

Prism, yellow

0.40 × 0.40 × 0.30 mm

Data collection

Enraf–Nonius CAD-4

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω/2θ scans

Absorption correction: ψ scan

(North *et al.*, 1968)

T_{min} = 0.999, *T_{max}* = 1.000

3150 measured reflections

1724 independent reflections

1463 reflections with *I* > 2σ(*I*)

R_{int} = 0.016

θ_{max} = 27.0°, θ_{min} = 2.8°

h = -9→4

k = -1→14

l = -12→12

2 standard reflections every 120 reflections

intensity decay: 1%

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.041

wR(*F*²) = 0.127

S = 1.08

1724 reflections

76 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0779P)^2 + 0.1315P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.50835 (7)	0.50319 (4)	0.13083 (5)	0.0515 (2)
S1	0.33563 (10)	0.44283 (7)	0.25236 (6)	0.0790 (3)
N1	0.4319 (2)	0.58738 (14)	-0.00532 (16)	0.0535 (4)
N2	0.6900 (3)	0.56638 (18)	0.21583 (18)	0.0670 (5)
C1	0.7415 (5)	0.5568 (3)	0.3657 (3)	0.0952 (9)
H1A	0.8411	0.5013	0.3838	0.143*
H1B	0.6352	0.5313	0.4096	0.143*
H1C	0.7821	0.6316	0.4024	0.143*
C2	0.8350 (4)	0.6172 (3)	0.1411 (3)	0.0814 (7)
H2A	0.8611	0.6952	0.1740	0.122*
H2B	0.7932	0.6190	0.0431	0.122*
H2C	0.9465	0.5708	0.1569	0.122*
C3	0.3911 (4)	0.7123 (2)	-0.0151 (3)	0.0779 (7)
H3A	0.4058	0.7454	0.0781	0.093*
H3B	0.4816	0.7496	-0.0679	0.093*
C4	0.2046 (5)	0.7387 (3)	-0.0806 (5)	0.1204 (13)
H4A	0.1842	0.7000	-0.1692	0.181*
H4B	0.1921	0.8216	-0.0940	0.181*
H4C	0.1139	0.7121	-0.0218	0.181*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0546 (3)	0.0576 (3)	0.0405 (3)	0.0050 (2)	-0.0029 (2)	0.00125 (18)
S1	0.0763 (4)	0.1040 (5)	0.0580 (4)	-0.0017 (3)	0.0128 (3)	0.0143 (3)
N1	0.0607 (9)	0.0518 (8)	0.0455 (8)	0.0091 (7)	-0.0047 (6)	0.0007 (6)
N2	0.0686 (11)	0.0823 (12)	0.0463 (8)	-0.0038 (9)	-0.0108 (8)	-0.0049 (8)
C1	0.099 (2)	0.130 (2)	0.0502 (12)	-0.0017 (18)	-0.0202 (13)	-0.0108 (14)
C2	0.0660 (13)	0.0974 (18)	0.0771 (15)	-0.0159 (13)	-0.0078 (11)	-0.0025 (14)
C3	0.0919 (17)	0.0540 (11)	0.0833 (15)	0.0127 (11)	-0.0097 (13)	-0.0028 (11)
C4	0.091 (2)	0.087 (2)	0.178 (4)	0.0302 (17)	-0.007 (2)	0.038 (2)

Geometric parameters (\AA , $^\circ$)

P1—N2	1.6325 (19)	C1—H1C	0.9600
P1—N1	1.6719 (16)	C2—H2A	0.9600

P1—N1 ⁱ	1.6856 (17)	C2—H2B	0.9600
P1—S1	1.9291 (9)	C2—H2C	0.9600
P1—P1 ⁱ	2.5143 (10)	C3—C4	1.450 (4)
N1—C3	1.460 (3)	C3—H3A	0.9700
N1—P1 ⁱ	1.6857 (17)	C3—H3B	0.9700
N2—C2	1.455 (3)	C4—H4A	0.9600
N2—C1	1.456 (3)	C4—H4B	0.9600
C1—H1A	0.9600	C4—H4C	0.9600
C1—H1B	0.9600		
N2—P1—N1	108.31 (10)	H1B—C1—H1C	109.5
N2—P1—N1 ⁱ	112.28 (10)	N2—C2—H2A	109.5
N1—P1—N1 ⁱ	83.02 (8)	N2—C2—H2B	109.5
N2—P1—S1	112.81 (8)	H2A—C2—H2B	109.5
N1—P1—S1	120.38 (7)	N2—C2—H2C	109.5
N1 ⁱ —P1—S1	116.69 (7)	H2A—C2—H2C	109.5
N2—P1—P1 ⁱ	117.58 (8)	H2B—C2—H2C	109.5
S1—P1—P1 ⁱ	129.60 (4)	C4—C3—N1	113.7 (2)
C3—N1—P1	131.38 (16)	C4—C3—H3A	108.8
C3—N1—P1 ⁱ	128.43 (17)	N1—C3—H3A	108.8
P1—N1—P1 ⁱ	96.98 (8)	C4—C3—H3B	108.8
C2—N2—C1	113.9 (2)	N1—C3—H3B	108.8
C2—N2—P1	120.47 (15)	H3A—C3—H3B	107.7
C1—N2—P1	124.5 (2)	C3—C4—H4A	109.5
N2—C1—H1A	109.5	C3—C4—H4B	109.5
N2—C1—H1B	109.5	H4A—C4—H4B	109.5
H1A—C1—H1B	109.5	C3—C4—H4C	109.5
N2—C1—H1C	109.5	H4A—C4—H4C	109.5
H1A—C1—H1C	109.5	H4B—C4—H4C	109.5

Symmetry code: (i) $-x+1, -y+1, -z$.