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# 1-Diphenylphosphanyl-2-(diphenylphosphoryl)-hydrazine

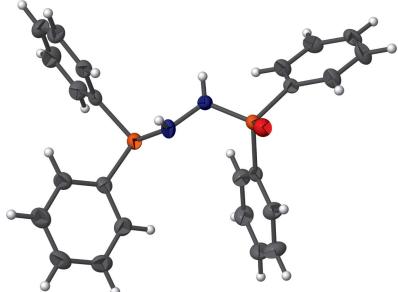
Martha Höhne, Bhaskar R. Aluri, Anke Spannenberg, Bernd H. Müller, Normen Peulecke and Uwe Rosenthal\*

Leibniz-Institut für Katalyse e. V. an der Universität Rostock, Albert-Einstein-Strasse 29a, 18059 Rostock, Germany.

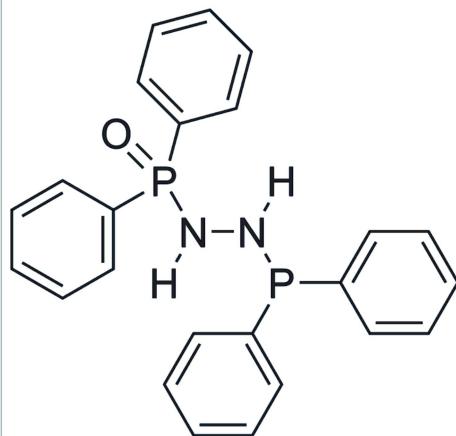
\*Correspondence e-mail: uwe.rosenthal@catalysis.de

The title compound,  $C_{24}H_{22}N_2OP_2$ , is an asymmetrically substituted hydrazine derivative bearing a phosphoryl and a phosphanyl substituent. The PNNP backbone has a torsion angle of  $-131.01(8)^\circ$ . In the crystal, molecules form centrosymmetric dimers by intermolecular N—H $\cdots$ O hydrogen bonds, which are further linked into a three-dimensional network by weak C—H $\cdots$ O and C—H $\cdots$  $\pi$  interactions.

## 3D view



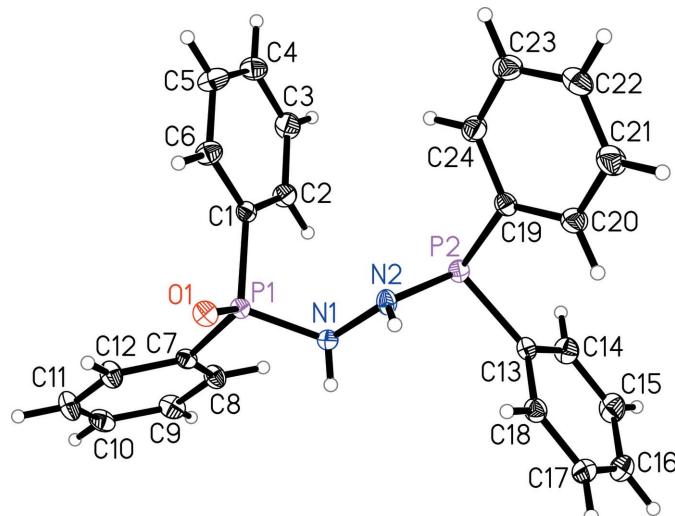
## Chemical scheme



## Structure description

The title compound  $C_{24}H_{22}N_2OP_2$  (Fig. 1) is an asymmetrically substituted hydrazine derivative containing a phosphoryl and phosphane entity. The PNNP backbone has a torsion angle of  $-131.01(8)^\circ$ . As a result of the asymmetrical substitution, the P—N bond lengths have significantly different values. Even if both P—N bond lengths are shortened when compared to the sum of the covalent radii calculated by Pyykkö (2015) [single:  $\Sigma r_{\text{cov}}(P—N) = 1.82 \text{ \AA}$ , double:  $\Sigma r_{\text{cov}}(P—N) = 1.62 \text{ \AA}$ ], the  $P^V—N$  distance [ $P1—N1 = 1.6561(11) \text{ \AA}$ ] is noticeably shorter than the  $P^{III—N}$  distance [ $P2—N2 = 1.7049(11) \text{ \AA}$ ]. The more pronounced reduction of  $P^V—N$  bond lengths of phosphoryl hydrazine entities [range from 1.6587(15) to 1.6989(10)  $\text{\AA}$ ; Gholivand *et al.*, 2012, 2016; Höhne *et al.*, 2018] in comparison to phosphane hydrazine  $P^{III—N}$  distances [range from 1.692(2) to 1.728(2)  $\text{\AA}$ ; Kriel *et al.*, 2010; Aluri *et al.*, 2010; Sushev *et al.*, 2008] is documented. The N—N distance within the hydrazine unit amounts to 1.4256(16)  $\text{\AA}$  and conforms to the sum of the covalent radii calculated by Pyykkö (2015) [ $\Sigma r_{\text{cov}}(N—N) = 1.42 \text{ \AA}$ ].

In the crystal, centrosymmetrically related molecules of the title compound are linked by pairs of intermolecular N—H $\cdots$ O hydrogen bonds (Table 1) forming dimers, which

**Figure 1**

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

are further linked by weak C—H···O and C—H··· $\pi$  interactions into a three-dimensional network.

### Synthesis and crystallization

A solution of NEt<sub>3</sub> (7.7 ml, 55.0 mmol) in THF was added to another solution of N<sub>2</sub>H<sub>4</sub>·HCl (0.685 g, 10.0 mmol) and Ph<sub>2</sub>PCl (3.7 ml, 20.0 mmol) in THF (20.0 ml). The mixture was stirred for 24 h at room temperature. Afterwards it was filtered; the solvent was removed in vacuum. 7.0 ml of toluene were added. The microcrystalline product was identified by NMR to be (Ph<sub>2</sub>P)<sub>2</sub>N—NH<sub>2</sub>. During the repeated attempts to crystallize (Ph<sub>2</sub>P)<sub>2</sub>N—NH<sub>2</sub>, the compound could have had some air contact, followed by rearrangement. The title compound was recrystallized from toluene.

### Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

### Funding information

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**Table 1**  
Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

*Cg1* and *Cg2* are the centroids of the C19–C24 and C7–C12 phenyl rings, respectively.

| <i>D</i> —H··· <i>A</i>               | <i>D</i> —H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> —H··· <i>A</i> |
|---------------------------------------|-------------|---------------|-----------------------|-------------------------|
| N2—H2B···O1 <sup>i</sup>              | 0.828 (18)  | 2.132 (18)    | 2.9357 (14)           | 163.6 (17)              |
| C10—H10A···O1 <sup>ii</sup>           | 0.95        | 2.50          | 3.2854 (19)           | 140                     |
| C15—H15A··· <i>Cg1</i> <sup>iii</sup> | 0.95        | 2.70          | 3.6125 (17)           | 162                     |
| C17—H17A··· <i>Cg2</i> <sup>iv</sup>  | 0.95        | 2.91          | 3.7171 (18)           | 144                     |

Symmetry codes: (i)  $-x + 1, -y + 1, -z$ ; (ii)  $x + 1, y, z$ ; (iii)  $-x + 1, -y, -z + 1$ ; (iv)  $x, y - 1, z$ .

**Table 2**  
Experimental details.

|  |  |
|--|--|
| Crystal data   | $C_{24}H_{22}N_2OP_2$  |
| Chemical formula   | 416.37   |
| $M_r$  | Triclinic, $P\bar{1}$  |
| Crystal system, space group  | 200  |
| Temperature (K)  | 8.4464 (4), 10.4163 (5), 13.4880 (6)                                   |
| $a, b, c$ (Å)  | 71.550 (4), 76.477 (4), 71.750 (4)                                     |
| $\alpha, \beta, \gamma$ ( $^\circ$ )                                       | 1057.04 (9)  |
| $V$ (Å $^3$ )  | 2  |
| Z  | Radiation type   |
|  | Mo $K\alpha$   |
| $\mu$ (mm $^{-1}$ )  | 0.22   |
| Crystal size (mm)  | 0.43 $\times$ 0.20 $\times$ 0.16                                       |
|  |  |
| Data collection  |  |
| Diffractometer   | Stoe IPDS II   |
| No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections | 18510, 5106, 3786  |
| $R_{\text{int}}$   | 0.034  |
| (sin $\theta/\lambda$ ) $_{\text{max}}$ (Å $^{-1}$ )                       | 0.661  |
|  |  |
| Refinement   |  |
| $R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , $S$                                  | 0.031, 0.075, 0.89   |
| No. of reflections   | 5106   |
| No. of parameters  | 270  |
| H-atom treatment   | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å $^{-3}$ )          | 0.38, -0.22  |

Computer programs: *X-Area* (Stoe & Cie, 2012), *XP* in *SHELXTL* and *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *publCIF* (Westrip, 2010).

# full crystallographic data

*IUCrData* (2018). **3**, x181784 [https://doi.org/10.1107/S2414314618017844]

## 1-Diphenylphosphanyl-2-(diphenylphosphoryl)hydrazine

Martha Höhne, Bhaskar R. Aluri, Anke Spannenberg, Bernd H. Müller, Normen Peulecke and Uwe Rosenthal

### 1-Diphenylphosphanyl-2-(diphenylphosphoryl)hydrazine

#### Crystal data

|                                 |   |
|---------------------------------|---|
| $C_{24}H_{22}N_2OP_2$           | $Z = 2$   |
| $M_r = 416.37$                  | $F(000) = 436$  |
| Triclinic, $P\bar{1}$           | $D_x = 1.308 \text{ Mg m}^{-3}$                         |
| $a = 8.4464 (4) \text{ \AA}$    | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| $b = 10.4163 (5) \text{ \AA}$   | Cell parameters from 7632 reflections                   |
| $c = 13.4880 (6) \text{ \AA}$   | $\theta = 2.1\text{--}29.7^\circ$                       |
| $\alpha = 71.550 (4)^\circ$     | $\mu = 0.22 \text{ mm}^{-1}$                            |
| $\beta = 76.477 (4)^\circ$      | $T = 200 \text{ K}$                                     |
| $\gamma = 71.750 (4)^\circ$     | Prism, colourless                                       |
| $V = 1057.04 (9) \text{ \AA}^3$ | $0.43 \times 0.20 \times 0.16 \text{ mm}$               |

#### Data collection

|  |   |
|--|---|
| Stoe IPDS II                             | 3786 reflections with $I > 2\sigma(I)$                              |
| diffractometer                           | $R_{\text{int}} = 0.034$  |
| Radiation source: fine-focus sealed tube | $\theta_{\text{max}} = 28.0^\circ, \theta_{\text{min}} = 2.1^\circ$ |
| Graphite monochromator                   | $h = -11 \rightarrow 10$  |
| $\omega$ scans                           | $k = -13 \rightarrow 13$  |
| 18510 measured reflections               | $l = -17 \rightarrow 17$  |
| 5106 independent reflections             |   |

#### Refinement

|                                 |  |
|---------------------------------|--|
| Refinement on $F^2$             | Hydrogen site location: mixed                        |
| Least-squares matrix: full      | H atoms treated by a mixture of independent          |
| $R[F^2 > 2\sigma(F^2)] = 0.031$ | and constrained refinement                           |
| $wR(F^2) = 0.075$               | $w = 1/[\sigma^2(F_o^2) + (0.0467P)^2]$              |
| $S = 0.89$                      | where $P = (F_o^2 + 2F_c^2)/3$                       |
| 5106 reflections                | $(\Delta/\sigma)_{\text{max}} = 0.001$               |
| 270 parameters                  | $\Delta\rho_{\text{max}} = 0.38 \text{ e \AA}^{-3}$  |
| 0 restraints                    | $\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$ |

#### 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.

**Refinement.** The N-bound hydrogen atoms H1 and H2B could be found from the difference Fourier map and were refined freely. All other H atoms were placed in idealized positions with  $d(\text{C---H}) = 0.95 \text{ \AA}$  and refined using a riding model with  $U_{\text{iso}}(\text{H})$  fixed at 1.2  $U_{\text{eq}}(\text{C})$ .

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|      | <i>x</i>     | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|---------------|---------------|----------------------------------|
| C1   | 0.48544 (17) | 0.69757 (13)  | 0.19890 (10)  | 0.0229 (3)                       |
| C2   | 0.5326 (2)   | 0.64375 (15)  | 0.29896 (11)  | 0.0315 (3)                       |
| H2A  | 0.6299       | 0.5676        | 0.3110        | 0.038*                           |
| C3   | 0.4392 (2)   | 0.70007 (17)  | 0.38115 (12)  | 0.0396 (4)                       |
| H3A  | 0.4725       | 0.6630        | 0.4494        | 0.048*                           |
| C4   | 0.2977 (2)   | 0.81011 (17)  | 0.36409 (13)  | 0.0416 (4)                       |
| H4A  | 0.2326       | 0.8481        | 0.4208        | 0.050*                           |
| C5   | 0.2506 (2)   | 0.86493 (18)  | 0.26515 (14)  | 0.0439 (4)                       |
| H5A  | 0.1534       | 0.9413        | 0.2536        | 0.053*                           |
| C6   | 0.34393 (19) | 0.80947 (15)  | 0.18232 (12)  | 0.0328 (3)                       |
| H6A  | 0.3111       | 0.8481        | 0.1140        | 0.039*                           |
| C7   | 0.80465 (17) | 0.66634 (13)  | 0.05663 (10)  | 0.0229 (3)                       |
| C8   | 0.93398 (19) | 0.59595 (15)  | 0.11728 (11)  | 0.0302 (3)                       |
| H8A  | 0.9210       | 0.5185        | 0.1762        | 0.036*                           |
| C9   | 1.08141 (19) | 0.63851 (17)  | 0.09194 (12)  | 0.0347 (3)                       |
| H9A  | 1.1696       | 0.5900        | 0.1334        | 0.042*                           |
| C10  | 1.10071 (19) | 0.75072 (17)  | 0.00700 (12)  | 0.0344 (3)                       |
| H10A | 1.2014       | 0.7805        | -0.0094       | 0.041*                           |
| C11  | 0.9744 (2)   | 0.82020 (17)  | -0.05451 (12) | 0.0382 (4)                       |
| H11A | 0.9888       | 0.8970        | -0.1136       | 0.046*                           |
| C12  | 0.82649 (19) | 0.77819 (15)  | -0.03034 (11) | 0.0319 (3)                       |
| H12A | 0.7400       | 0.8258        | -0.0732       | 0.038*                           |
| C13  | 0.61345 (17) | 0.11965 (13)  | 0.30235 (10)  | 0.0246 (3)                       |
| C14  | 0.6910 (2)   | 0.02742 (16)  | 0.38775 (12)  | 0.0351 (3)                       |
| H14A | 0.6779       | 0.0565        | 0.4499        | 0.042*                           |
| C15  | 0.7867 (2)   | -0.10579 (17) | 0.38350 (13)  | 0.0429 (4)                       |
| H15A | 0.8378       | -0.1679       | 0.4428        | 0.052*                           |
| C16  | 0.8082 (2)   | -0.14876 (16) | 0.29358 (14)  | 0.0414 (4)                       |
| H16A | 0.8756       | -0.2398       | 0.2902        | 0.050*                           |
| C17  | 0.7314 (2)   | -0.05915 (15) | 0.20848 (12)  | 0.0365 (3)                       |
| H17A | 0.7451       | -0.0891       | 0.1467        | 0.044*                           |
| C18  | 0.63471 (19) | 0.07405 (14)  | 0.21248 (11)  | 0.0287 (3)                       |
| H18A | 0.5823       | 0.1349        | 0.1534        | 0.034*                           |
| C19  | 0.28166 (18) | 0.29918 (14)  | 0.33548 (10)  | 0.0261 (3)                       |
| C20  | 0.2203 (2)   | 0.19537 (16)  | 0.32568 (12)  | 0.0354 (3)                       |
| H20A | 0.2967       | 0.1167        | 0.3042        | 0.042*                           |
| C21  | 0.0494 (2)   | 0.20513 (18)  | 0.34677 (14)  | 0.0419 (4)                       |
| H21A | 0.0093       | 0.1337        | 0.3390        | 0.050*                           |
| C22  | -0.0634 (2)  | 0.31765 (17)  | 0.37906 (12)  | 0.0377 (4)                       |
| H22A | -0.1808      | 0.3240        | 0.3936        | 0.045*                           |
| C23  | -0.0042 (2)  | 0.42118 (16)  | 0.39012 (12)  | 0.0384 (4)                       |
| H23A | -0.0811      | 0.4987        | 0.4128        | 0.046*                           |
| C24  | 0.1660 (2)   | 0.41228 (15)  | 0.36827 (12)  | 0.0339 (3)                       |
| H24A | 0.2052       | 0.4844        | 0.3757        | 0.041*                           |
| N1   | 0.65219 (16) | 0.45605 (12)  | 0.14559 (9)   | 0.0252 (2)                       |

|     |              |              |              |             |
|-----|--------------|--------------|--------------|-------------|
| N2  | 0.50757 (15) | 0.40184 (11) | 0.18533 (9)  | 0.0259 (2)  |
| O1  | 0.50978 (12) | 0.68341 (10) | -0.00130 (7) | 0.0275 (2)  |
| P1  | 0.60334 (4)  | 0.62701 (3)  | 0.09005 (3)  | 0.02070 (8) |
| P2  | 0.50410 (5)  | 0.29827 (4)  | 0.31182 (3)  | 0.02479 (9) |
| H1  | 0.732 (2)    | 0.4119 (19)  | 0.1144 (15)  | 0.044 (5)*  |
| H2B | 0.482 (2)    | 0.3803 (18)  | 0.1384 (14)  | 0.036 (5)*  |

*Atomic displacement parameters ( $\text{\AA}^2$ )*

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$      | $U^{13}$      | $U^{23}$      |
|-----|--------------|--------------|--------------|---------------|---------------|---------------|
| C1  | 0.0239 (7)   | 0.0243 (6)   | 0.0238 (6)   | -0.0102 (5)   | -0.0020 (5)   | -0.0081 (5)   |
| C2  | 0.0354 (8)   | 0.0317 (7)   | 0.0270 (7)   | -0.0057 (6)   | -0.0070 (6)   | -0.0082 (5)   |
| C3  | 0.0557 (11)  | 0.0402 (8)   | 0.0244 (7)   | -0.0137 (8)   | -0.0051 (7)   | -0.0098 (6)   |
| C4  | 0.0500 (10)  | 0.0414 (8)   | 0.0338 (8)   | -0.0118 (7)   | 0.0067 (7)    | -0.0200 (7)   |
| C5  | 0.0395 (10)  | 0.0407 (9)   | 0.0474 (9)   | 0.0040 (7)    | -0.0058 (8)   | -0.0210 (7)   |
| C6  | 0.0328 (8)   | 0.0334 (7)   | 0.0328 (7)   | -0.0032 (6)   | -0.0082 (6)   | -0.0123 (6)   |
| C7  | 0.0221 (7)   | 0.0266 (6)   | 0.0212 (6)   | -0.0075 (5)   | -0.0023 (5)   | -0.0075 (5)   |
| C8  | 0.0279 (8)   | 0.0342 (7)   | 0.0258 (6)   | -0.0087 (6)   | -0.0059 (6)   | -0.0022 (5)   |
| C9  | 0.0244 (8)   | 0.0465 (9)   | 0.0335 (7)   | -0.0072 (7)   | -0.0087 (6)   | -0.0097 (6)   |
| C10 | 0.0252 (8)   | 0.0479 (9)   | 0.0358 (8)   | -0.0172 (7)   | 0.0020 (6)    | -0.0159 (7)   |
| C11 | 0.0368 (9)   | 0.0422 (8)   | 0.0333 (8)   | -0.0204 (7)   | -0.0036 (7)   | 0.0021 (6)    |
| C12 | 0.0288 (8)   | 0.0362 (7)   | 0.0286 (7)   | -0.0112 (6)   | -0.0088 (6)   | 0.0000 (6)    |
| C13 | 0.0242 (7)   | 0.0254 (6)   | 0.0246 (6)   | -0.0102 (5)   | -0.0031 (5)   | -0.0036 (5)   |
| C14 | 0.0407 (9)   | 0.0359 (7)   | 0.0277 (7)   | -0.0094 (7)   | -0.0106 (6)   | -0.0038 (6)   |
| C15 | 0.0464 (10)  | 0.0344 (8)   | 0.0391 (9)   | -0.0046 (7)   | -0.0149 (7)   | 0.0029 (6)    |
| C16 | 0.0439 (10)  | 0.0265 (7)   | 0.0462 (9)   | -0.0045 (7)   | -0.0028 (7)   | -0.0065 (6)   |
| C17 | 0.0447 (10)  | 0.0316 (7)   | 0.0334 (8)   | -0.0114 (7)   | 0.0008 (7)    | -0.0122 (6)   |
| C18 | 0.0328 (8)   | 0.0293 (7)   | 0.0237 (6)   | -0.0099 (6)   | -0.0041 (6)   | -0.0046 (5)   |
| C19 | 0.0291 (7)   | 0.0288 (6)   | 0.0193 (6)   | -0.0092 (6)   | -0.0026 (5)   | -0.0039 (5)   |
| C20 | 0.0316 (8)   | 0.0380 (8)   | 0.0405 (8)   | -0.0126 (6)   | 0.0012 (7)    | -0.0167 (7)   |
| C21 | 0.0352 (9)   | 0.0490 (9)   | 0.0476 (9)   | -0.0188 (7)   | -0.0009 (7)   | -0.0171 (8)   |
| C22 | 0.0266 (8)   | 0.0475 (9)   | 0.0308 (7)   | -0.0078 (7)   | -0.0032 (6)   | -0.0016 (6)   |
| C23 | 0.0347 (9)   | 0.0360 (8)   | 0.0339 (8)   | 0.0006 (7)    | -0.0035 (7)   | -0.0058 (6)   |
| C24 | 0.0383 (9)   | 0.0288 (7)   | 0.0324 (7)   | -0.0066 (6)   | -0.0052 (6)   | -0.0073 (6)   |
| N1  | 0.0251 (6)   | 0.0233 (5)   | 0.0266 (6)   | -0.0070 (5)   | 0.0006 (5)    | -0.0080 (4)   |
| N2  | 0.0336 (7)   | 0.0259 (5)   | 0.0226 (5)   | -0.0143 (5)   | -0.0060 (5)   | -0.0045 (4)   |
| O1  | 0.0285 (5)   | 0.0326 (5)   | 0.0238 (5)   | -0.0090 (4)   | -0.0077 (4)   | -0.0070 (4)   |
| P1  | 0.02160 (18) | 0.02251 (16) | 0.01936 (15) | -0.00718 (13) | -0.00382 (12) | -0.00538 (12) |
| P2  | 0.0297 (2)   | 0.02590 (17) | 0.02158 (16) | -0.01021 (14) | -0.00453 (14) | -0.00654 (13) |

*Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

|        |             |          |           |
|--------|-------------|----------|-----------|
| C1—C6  | 1.3874 (19) | C14—C15  | 1.381 (2) |
| C1—C2  | 1.3883 (18) | C14—H14A | 0.9500    |
| C1—P1  | 1.7967 (13) | C15—C16  | 1.376 (2) |
| C2—C3  | 1.382 (2)   | C15—H15A | 0.9500    |
| C2—H2A | 0.9500      | C16—C17  | 1.378 (2) |
| C3—C4  | 1.378 (2)   | C16—H16A | 0.9500    |

|            |             |              |             |
|------------|-------------|--------------|-------------|
| C3—H3A     | 0.9500      | C17—C18      | 1.383 (2)   |
| C4—C5      | 1.374 (2)   | C17—H17A     | 0.9500      |
| C4—H4A     | 0.9500      | C18—H18A     | 0.9500      |
| C5—C6      | 1.384 (2)   | C19—C20      | 1.389 (2)   |
| C5—H5A     | 0.9500      | C19—C24      | 1.395 (2)   |
| C6—H6A     | 0.9500      | C19—P2       | 1.8283 (15) |
| C7—C12     | 1.3914 (19) | C20—C21      | 1.382 (2)   |
| C7—C8      | 1.3916 (19) | C20—H20A     | 0.9500      |
| C7—P1      | 1.7953 (14) | C21—C22      | 1.379 (2)   |
| C8—C9      | 1.384 (2)   | C21—H21A     | 0.9500      |
| C8—H8A     | 0.9500      | C22—C23      | 1.382 (2)   |
| C9—C10     | 1.375 (2)   | C22—H22A     | 0.9500      |
| C9—H9A     | 0.9500      | C23—C24      | 1.378 (2)   |
| C10—C11    | 1.379 (2)   | C23—H23A     | 0.9500      |
| C10—H10A   | 0.9500      | C24—H24A     | 0.9500      |
| C11—C12    | 1.386 (2)   | N1—N2        | 1.4256 (16) |
| C11—H11A   | 0.9500      | N1—P1        | 1.6561 (11) |
| C12—H12A   | 0.9500      | N1—H1        | 0.80 (2)    |
| C13—C18    | 1.3915 (18) | N2—P2        | 1.7049 (12) |
| C13—C14    | 1.3918 (19) | N2—H2B       | 0.828 (18)  |
| C13—P2     | 1.8304 (14) | O1—P1        | 1.4813 (9)  |
| <br>       |             |              |             |
| C6—C1—C2   | 119.11 (13) | C14—C15—H15A | 119.9       |
| C6—C1—P1   | 119.08 (10) | C15—C16—C17  | 119.71 (15) |
| C2—C1—P1   | 121.82 (10) | C15—C16—H16A | 120.1       |
| C3—C2—C1   | 120.48 (14) | C17—C16—H16A | 120.1       |
| C3—C2—H2A  | 119.8       | C16—C17—C18  | 120.43 (14) |
| C1—C2—H2A  | 119.8       | C16—C17—H17A | 119.8       |
| C4—C3—C2   | 119.92 (14) | C18—C17—H17A | 119.8       |
| C4—C3—H3A  | 120.0       | C17—C18—C13  | 120.55 (13) |
| C2—C3—H3A  | 120.0       | C17—C18—H18A | 119.7       |
| C5—C4—C3   | 120.08 (14) | C13—C18—H18A | 119.7       |
| C5—C4—H4A  | 120.0       | C20—C19—C24  | 118.04 (14) |
| C3—C4—H4A  | 120.0       | C20—C19—P2   | 125.26 (11) |
| C4—C5—C6   | 120.32 (15) | C24—C19—P2   | 116.68 (11) |
| C4—C5—H5A  | 119.8       | C21—C20—C19  | 120.75 (14) |
| C6—C5—H5A  | 119.8       | C21—C20—H20A | 119.6       |
| C5—C6—C1   | 120.08 (14) | C19—C20—H20A | 119.6       |
| C5—C6—H6A  | 120.0       | C22—C21—C20  | 120.52 (15) |
| C1—C6—H6A  | 120.0       | C22—C21—H21A | 119.7       |
| C12—C7—C8  | 119.27 (13) | C20—C21—H21A | 119.7       |
| C12—C7—P1  | 117.66 (10) | C21—C22—C23  | 119.42 (15) |
| C8—C7—P1   | 122.95 (10) | C21—C22—H22A | 120.3       |
| C9—C8—C7   | 120.12 (13) | C23—C22—H22A | 120.3       |
| C9—C8—H8A  | 119.9       | C24—C23—C22  | 120.17 (14) |
| C7—C8—H8A  | 119.9       | C24—C23—H23A | 119.9       |
| C10—C9—C8  | 120.22 (14) | C22—C23—H23A | 119.9       |
| C10—C9—H9A | 119.9       | C23—C24—C19  | 121.09 (14) |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C8—C9—H9A       | 119.9        | C23—C24—H24A    | 119.5        |
| C9—C10—C11      | 120.22 (14)  | C19—C24—H24A    | 119.5        |
| C9—C10—H10A     | 119.9        | N2—N1—P1        | 112.75 (9)   |
| C11—C10—H10A    | 119.9        | N2—N1—H1        | 117.6 (14)   |
| C10—C11—C12     | 120.14 (14)  | P1—N1—H1        | 114.7 (14)   |
| C10—C11—H11A    | 119.9        | N1—N2—P2        | 114.25 (9)   |
| C12—C11—H11A    | 119.9        | N1—N2—H2B       | 110.2 (12)   |
| C11—C12—C7      | 120.01 (13)  | P2—N2—H2B       | 121.8 (12)   |
| C11—C12—H12A    | 120.0        | O1—P1—N1        | 119.35 (6)   |
| C7—C12—H12A     | 120.0        | O1—P1—C7        | 111.77 (6)   |
| C18—C13—C14     | 118.22 (13)  | N1—P1—C7        | 102.73 (6)   |
| C18—C13—P2      | 124.11 (10)  | O1—P1—C1        | 110.99 (6)   |
| C14—C13—P2      | 117.48 (10)  | N1—P1—C1        | 102.73 (6)   |
| C15—C14—C13     | 120.97 (14)  | C7—P1—C1        | 108.36 (6)   |
| C15—C14—H14A    | 119.5        | N2—P2—C19       | 96.79 (6)    |
| C13—C14—H14A    | 119.5        | N2—P2—C13       | 106.30 (6)   |
| C16—C15—C14     | 120.11 (14)  | C19—P2—C13      | 103.11 (6)   |
| C16—C15—H15A    | 119.9        |                 |              |
| <br>            |              |                 |              |
| C6—C1—C2—C3     | 0.5 (2)      | C22—C23—C24—C19 | -0.4 (2)     |
| P1—C1—C2—C3     | -179.87 (12) | C20—C19—C24—C23 | -0.1 (2)     |
| C1—C2—C3—C4     | 0.3 (2)      | P2—C19—C24—C23  | -178.73 (11) |
| C2—C3—C4—C5     | -0.8 (3)     | P1—N1—N2—P2     | -131.01 (8)  |
| C3—C4—C5—C6     | 0.5 (3)      | N2—N1—P1—O1     | -59.52 (11)  |
| C4—C5—C6—C1     | 0.3 (3)      | N2—N1—P1—C7     | 176.20 (9)   |
| C2—C1—C6—C5     | -0.8 (2)     | N2—N1—P1—C1     | 63.73 (10)   |
| P1—C1—C6—C5     | 179.58 (13)  | C12—C7—P1—O1    | 21.64 (13)   |
| C12—C7—C8—C9    | 1.0 (2)      | C8—C7—P1—O1     | -162.43 (11) |
| P1—C7—C8—C9     | -174.91 (11) | C12—C7—P1—N1    | 150.79 (11)  |
| C7—C8—C9—C10    | 0.2 (2)      | C8—C7—P1—N1     | -33.28 (13)  |
| C8—C9—C10—C11   | -1.0 (2)     | C12—C7—P1—C1    | -100.97 (11) |
| C9—C10—C11—C12  | 0.7 (2)      | C8—C7—P1—C1     | 74.96 (12)   |
| C10—C11—C12—C7  | 0.5 (2)      | C6—C1—P1—O1     | -11.40 (13)  |
| C8—C7—C12—C11   | -1.3 (2)     | C2—C1—P1—O1     | 169.00 (11)  |
| P1—C7—C12—C11   | 174.78 (12)  | C6—C1—P1—N1     | -140.06 (11) |
| C18—C13—C14—C15 | 0.1 (2)      | C2—C1—P1—N1     | 40.34 (13)   |
| P2—C13—C14—C15  | -175.06 (13) | C6—C1—P1—C7     | 111.69 (12)  |
| C13—C14—C15—C16 | 0.7 (3)      | C2—C1—P1—C7     | -67.91 (13)  |
| C14—C15—C16—C17 | -1.1 (3)     | N1—N2—P2—C19    | 165.14 (9)   |
| C15—C16—C17—C18 | 0.7 (3)      | N1—N2—P2—C13    | -89.02 (10)  |
| C16—C17—C18—C13 | 0.0 (2)      | C20—C19—P2—N2   | 97.04 (13)   |
| C14—C13—C18—C17 | -0.4 (2)     | C24—C19—P2—N2   | -84.50 (11)  |
| P2—C13—C18—C17  | 174.35 (12)  | C20—C19—P2—C13  | -11.50 (14)  |
| C24—C19—C20—C21 | 0.7 (2)      | C24—C19—P2—C13  | 166.97 (10)  |
| P2—C19—C20—C21  | 179.15 (12)  | C18—C13—P2—N2   | -21.59 (14)  |
| C19—C20—C21—C22 | -0.7 (2)     | C14—C13—P2—N2   | 153.23 (11)  |
| C20—C21—C22—C23 | 0.1 (2)      | C18—C13—P2—C19  | 79.63 (13)   |
| C21—C22—C23—C24 | 0.5 (2)      | C14—C13—P2—C19  | -105.55 (12) |

*Hydrogen-bond geometry (Å, °)*

Cg1 and Cg2 are the centroids of the C19–C24 and C7–C12 phenyl rings, respectively.

| $D\text{--H}\cdots A$         | $D\text{--H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D\text{--H}\cdots A$ |
|-------------------------------|---------------|--------------------|-------------|-----------------------|
| N2—H2B···O1 <sup>i</sup>      | 0.828 (18)    | 2.132 (18)         | 2.9357 (14) | 163.6 (17)            |
| C10—H10A···O1 <sup>ii</sup>   | 0.95          | 2.50               | 3.2854 (19) | 140                   |
| C15—H15A···Cg1 <sup>iii</sup> | 0.95          | 2.70               | 3.6125 (17) | 162                   |
| C17—H17A···Cg2 <sup>iv</sup>  | 0.95          | 2.91               | 3.7171 (18) | 144                   |

Symmetry codes: (i)  $-x+1, -y+1, -z$ ; (ii)  $x+1, y, z$ ; (iii)  $-x+1, -y, -z+1$ ; (iv)  $x, y-1, z$ .