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Dicarbon­yl{[(E,E)-(2,3,4,5-η)-6-meth­­oxy-6-oxo-2,4-hexa­dien­yl]tri­phenyl­phospho­nium}(tri­phenyl­phosphane-κP)iron(1+) hexa­fluorido­phosphate

Yuzhi Ma

Department of Chemistry, Marquette University, Milwaukee, WI

Sergey Lindeman

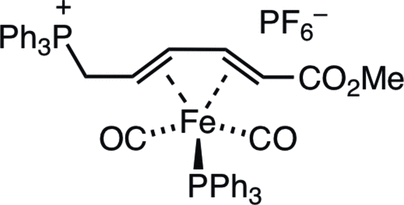
Department of Chemistry, Marquette University, Milwaukee, WI

William A. Donaldson

Department of Chemistry, Marquette University, Milwaukee, WI

In the title compound, [Fe(C25H24O2P)(C18H15P)(CO)2]PF6, the Fe atom adopts a square-based pyramidal coordination geometry with the carbonyl groups and the two C=C bonds of the diene defining the basal sites and the phosphane ligand the apical position. The diene ligand has an *E,E* geometry, with the phospho­nium fragment pointed away from the Fe atom. The crystal structure displays C—H⋯F and C—H⋯O hydrogen bonding. The PF6− anion is rotationally disordered over four orientations.

**Keywords:**crystal structure; 2*E*,4*E*-dienyl­phospho­nium; (penta­dien­yl)iron cation; Fe(CO)2PPh3.



# Structure description

Acyclic (dien­yl)iron cations and acyclic (diene)iron complexes have been utilized in organic synthesis as the coordination to iron serves as a protecting group for the diene against oxidation, reduction and cyclo­addition (Cox & Ley, 1998[[Cox, L. R. & Ley, S. V. (1998). Chem. Rev. 27, 301-314.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB13); Donaldson, 2000[[Donaldson, W. A. (2000). Curr. Org. Chem. 4, 837-868.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB4); Donaldson & Chaudhury, 2009[[Donaldson, W. A. & Chaudhury, S. (2009). Eur. J. Org. Chem. pp. 3831-3843.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB5); Grée, 1989[[Grée, R. (1989). Synthesis, pp. 341-355.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB7); Iwata & Takemoto, 1996[[Iwata, C. & Takemoto, Y. (1996). Chem. Commun. pp. 2497-2504.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB11)). The reaction of acyclic tricarbon­yl(dien­yl)iron cations with phosphines results in the formation of iron-complexed dienyl­phospho­nium salts (McArdle & Sherlock, 1978[[McArdle, P. & Sherlock, H. (1978). J. Chem. Soc. Dalton Trans. pp. 1678-1682.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB12); Salzer & Hafner, 1983[[Salzer, A. & Hafner, A. (1983). Helv. Chim. Acta, 66, 1774-1785.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB14)). It was eventually demonstrated that this reaction proceeds *via* kinetic nucleophilic attack on the *cisoid*form of the cation, and that this C—P bond formation is reversible in certain cases (Donaldson *et al.*, 1994[[Donaldson, W. A., Shang, L. & Rogers, R. D. (1994). Organometallics, 13, 6-7.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB17), 1995[[Donaldson, W. A., Shang, L., Ramaswamy, M., Droste, C. A., Tao, C. & Bennett, D. W. (1995). Organometallics, 14, 5119-5126.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB6)). These dienyl­phospho­nium salts have been utilized in Wittig-type olefination reactions (Hafner *et al.* 1983[[Hafner, A., Bieri, J. H., Prewo, R., von Philipsborn, W. & Salzer, A. (1983). Angew. Chem. Int. Ed. Engl. 22, 713-715.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB8)). To our knowledge, there is only one report of the reaction of a dicarbon­yl(tri­phenyl­phosphine)-ligated (penta­dien­yl)iron cation with tri­phenyl­phosphine (Chaudhury *et al.*, 2007[[Chaudhury, S., Li, S., Bennett, D. W., Siddiquee, T., Haworth, D. T. & Donaldson, W. A. (2007). Organometallics, 26, 5295-5303.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB2)). In a continuation of this work, we present here the synthesis and crystal structure of a 2*E*,4*E*-dienyl­phospho­nium salt complexed to an Fe(CO)2PPh3 fragment with hexa­fluorido­phosphate counter-ions.

The title compound (Fig. 1[[link]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#FIG1)) consists of a (*trans*,*trans*-dien­yl)phospho­nium ligand coordinated to iron, with the phospho­nium group orientated distal to the metal atom. The complex exhibits the typical square-based pyramidal structure of (diene)Fe(CO)2*L* complexes, with the tri­phenyl­phosphine ligand oriented in the axial position. Crystal structures of other acyclic (diene)Fe(CO)2*L* complexes exhibit both axial (Howell *et al.*, 1994[[Howell, J. A. S., Squibb, A. D., Bell, A. G., McArdle, P., Cunningham, D., Goldschmidt, Z., Gottlieb, H. E., Hezroni-Langerman, D. & Gree, R. (1994). Organometallics, 13, 4336-4351.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB10); Zutin *et al.*, 2001[[Zutin, K., Nogueira, V. M., Mauro, A. E., Melnikov, P. & Iluykhin, A. (2001). Polyhedron, 20, 1011-1016.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB16)) and basal (Howell *et al.*, 1996[[Howell, J. A. S., Bell, A. G., O'Leary, P. J., Stephenson, G. R., Hastings, M., Howard, P. W., Owen, D. A., Whitehead, A. J., McArdle, P. & Cunningham, D. (1996). Organometallics, 15, 4247-4257.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB9)) phosphine orientations. The crystal structure (Fig. 2[[link]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#FIG2)) exhibits several inter­actions between phenyl hydrogen atoms and fluorine atoms of the PF6−anion which are within the range of C—H⋯F hydrogen bonds (2.40–2.56 Å). C—H⋯F and C—H⋯O hydrogen-bond geometries are summarized in Table 1[[link]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#TABLE1).

**Table 1.** Hydrogen-bond geometry (Å, °)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| ***D*—H⋯*A*** | ***D*—H** | **H⋯*A*** | ***D*⋯*A*** | ***D*—H⋯*A*** |
| C26—H26⋯F1 | 0.95 | 2.43 | 3.1353 (15) | 131 |
| C28—H28⋯F5*B*i | 0.95 | 2.49 | 3.322 (8) | 146 |
| C28—H28⋯F5*C*i | 0.95 | 2.55 | 3.320 (15) | 139 |
| C32—H32⋯O4ii | 0.95 | 2.56 | 3.3659 (16) | 143 |
| C34—H34⋯F1 | 0.95 | 2.41 | 3.1349 (16) | 133 |
| C40—H40⋯F2iii | 0.95 | 2.40 | 3.2586 (16) | 150 |

3 1 1 3 1 1

Symmetry codes: (i) (ii)

(iii)

|  |
| --- |
|  |
| [[[Figure 1]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/hb4240fig1.html)](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/hb4240fig1.html)  [**Figure 1**](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/hb4240fig1.html)  A view of title compound showing 50% displacement ellipsoids and H atoms omitted for clarity. |

|  |  |
| --- | --- |
| [[[Figure 2]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/hb4240fig2.html)](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/hb4240fig2.html) | [**Figure 2**](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/hb4240fig2.html)  Packing diagram of the title compound. |

# Synthesis and crystallization

To a solution of dicarbon­yl(1-meth­oxy­carbonyl­penta­dien­yl)(tri­phenyl­phosphine)iron(1+) hexa­fluoro­phosphate (100 mg, 0.155 mmol) in CH2Cl2 (20 ml) was added tri­phenyl­phosphine (61 mg, 0.23 mmol). The reaction mixture was stirred for 24 h, and the solvent evaporated. The residue was taken up in a minimal amount of CH2Cl2 and diethyl ether was slowly added until the mixture became cloudy. After standing for 24 h, the yellow blocks of the title compound that had formed were collected (121 mg, 86%).

# Refinement

The crystal data, data collection and structure refinement details are summarized in Table 2[[link]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#TABLE2). The PF6− anion is rotationally disordered over four sets of sites (occupancy ratio 0.43/0.37/0.13/0.07).

**Table 2** Experimental details

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| |  |  | | --- | --- | | Crystal data |  | | Chemical formula | [Fe(C25H24O2P)(C18H15P)(CO)2]PF6 | | *M*r | 906.52 | | Crystal system, space group | Monoclinic, *P*21/*n* | | Temperature (K) | 100 | | *a*, *b*, *c* (Å) | 9.10074 (14), 17.7897 (3), 24.9408 (3) | | β (°) | 97.7826 (14) | | *V* (Å3) | 4000.72 (10) | | *Z* | 4 | | Radiation type | Mo *K*α | | μ (mm−1) | 0.57 | | Crystal size (mm) | 0.23 × 0.12 × 0.1 | | Data collection |  | | Diffractometer | Oxford Diffraction SuperNova, Dual, Cu at zero, Atlas | | Absorption correction | Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010[[Oxford Diffraction (2010). CrysAlis PRO. Oxford Diffraction Ltd, Yarnton, England.]](http://iucrdata.iucr.org/x/issues/2018/06/00/hb4240/index.html#BB18)) | | *T*min, *T*max | 0.830, 1.000 | | No. of measured, independent and observed [*I* > 2σ(*I*)] reflections | 79564, 14181, 11299 | | *R*int | 0.032 | | (sin θ/λ)max (Å−1) | 0.763 | | Refinement |  | | *R*[*F*2 > 2σ(*F*2)], *wR*(*F*2), *S* | 0.034, 0.095, 1.10 | | No. of reflections | 14181 | | No. of parameters | 573 | | No. of restraints | 60 | | H-atom treatment | H atoms treated by a mixture of independent and constrained refinement | | Δρmax, Δρmin (e Å−3) | 1.00, −0.68 | |

# 

# Structural data

CCDC reference: [1850684](http://scripts.iucr.org/cgi-bin/cr.cgi?rm=csd&csdid=1850684)

Crystal structure: contains datablock I. DOI: [https://doi.org//10.1107/S2414314618009021/hb4240sup1.cif](https://doi.org/10.1107/S2414314618009021/hb4240sup1.cif)

Structure factors: contains datablock I. DOI: [https://doi.org//10.1107/S2414314618009021/hb4240Isup2.hkl](https://doi.org/10.1107/S2414314618009021/hb4240Isup2.hkl)

[checkCIF report](http://scripts.iucr.org/cgi-bin/paper?hb4240&checkcif=yes)

** Full crystallographic data**

**Crystal data**

|  |  |
| --- | --- |
| [Fe(C25H24O2P)(C18H15P)(CO)2]PF6 | *F*(000) = 1864 |
| *Mr* = 906.52 | *D*x = 1.505 Mg m−3 |
| Monoclinic, *P*21/*n* | Mo *K*α radiation, λ = 0.71073 Å |
| *a* = 9.10074 (14) Å | Cell parameters from 37568 reflections |
| *b* = 17.7897 (3) Å | θ = 3.3–32.8° |
| *c* = 24.9408 (3) Å | µ = 0.57 mm−1 |
| β = 97.7826 (14)° | *T* = 100 K |
| *V* = 4000.72 (10) Å3 | Block, yellow |
| *Z* = 4 | 0.23 × 0.12 × 0.1 mm |

# Data collection

|  |  |
| --- | --- |
| Oxford Diffraction SuperNova, Dual, Cu at zero, Atlas  diffractometer | 14181 independent reflections |
| Radiation source: SuperNova (Mo) X-ray Source | 11299 reflections with *I* > 2σ(*I*) |
| Mirror monochromator | *R*int = 0.032 |
| Detector resolution: 10.3756 pixels mm-1 | θmax = 32.8°, θmin = 3.3° |
| ω scans | *h* = −13→13 |
| Absorption correction: multi-scan  (CrysAlis PRO; Oxford Diffraction, 2010) | *k* = −26→27 |
| *T*min = 0.830, *T*max = 1.000 | *l* = −36→37 |
| 79564 measured reflections |  |

# Refinement

|  |  |
| --- | --- |
| Refinement on *F*2 | Primary atom site location: iterative |
| Least-squares matrix: full | Hydrogen site location: mixed |
| *R*[*F*2 > 2σ(*F*2)] = 0.034 | H atoms treated by a mixture of independent and constrained refinement |
| *wR*(*F*2) = 0.095 | *w* = 1/[σ2(*F*o2) + (0.0494*P*)2 + 0.8274*P*]  where *P* = (*F*o2 + 2*F*c2)/3 |
| *S* = 1.10 | (Δ/σ)max = 0.002 |
| 14181 reflections | Δρmax = 1.00 e Å−3 |
| 573 parameters | Δρmin = −0.68 e Å−3 |
| 60 restraints |  |

# 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 (Å2)

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | *x* | *y* | *z* | *U*iso\*/*U*eq | Occ. (<1) |
| Fe1 | 0.50960 (2) | 0.18533 (2) | 0.15093 (2) | 0.01166 (4) |  |
| P1 | 0.53777 (3) | 0.28527 (2) | 0.09909 (2) | 0.01150 (6) |  |
| P2 | 0.67576 (3) | 0.28331 (2) | 0.33137 (2) | 0.01235 (6) |  |
| O1 | 0.33714 (11) | 0.08143 (6) | 0.07598 (4) | 0.0254 (2) |  |
| O2 | 0.22620 (11) | 0.23511 (7) | 0.18090 (4) | 0.0293 (2) |  |
| O3 | 0.85441 (11) | 0.12133 (6) | 0.07077 (4) | 0.0243 (2) |  |
| O4 | 0.67251 (10) | 0.04156 (5) | 0.08681 (4) | 0.02058 (18) |  |
| C1 | 0.40987 (13) | 0.12215 (7) | 0.10423 (5) | 0.0166 (2) |  |
| C2 | 0.33740 (14) | 0.21459 (8) | 0.17000 (5) | 0.0180 (2) |  |
| C3 | 0.76111 (13) | 0.10278 (7) | 0.09825 (5) | 0.0175 (2) |  |
| C4 | 0.72672 (13) | 0.14296 (7) | 0.14662 (5) | 0.0155 (2) |  |
| H4 | 0.7993 (18) | 0.1815 (9) | 0.1577 (7) | 0.019\* |  |
| C5 | 0.65260 (13) | 0.10463 (7) | 0.18571 (5) | 0.0156 (2) |  |
| H5 | 0.6340 (17) | 0.0511 (10) | 0.1824 (6) | 0.019\* |  |
| C6 | 0.60214 (13) | 0.14789 (7) | 0.22669 (5) | 0.0152 (2) |  |
| H6 | 0.5415 (17) | 0.1248 (9) | 0.2520 (7) | 0.018\* |  |
| C7 | 0.62429 (13) | 0.22720 (7) | 0.22568 (4) | 0.0141 (2) |  |
| H7 | 0.7171 (17) | 0.2484 (9) | 0.2157 (6) | 0.017\* |  |
| C8 | 0.55786 (13) | 0.27530 (7) | 0.26610 (4) | 0.0149 (2) |  |
| H8A | 0.461176 | 0.253617 | 0.272032 | 0.018\* |  |
| H8B | 0.539080 | 0.326234 | 0.250704 | 0.018\* |  |
| C9 | 0.60530 (12) | 0.27239 (7) | 0.03350 (4) | 0.0139 (2) |  |
| C10 | 0.58330 (13) | 0.20434 (7) | 0.00597 (5) | 0.0165 (2) |  |
| H10 | 0.536751 | 0.163867 | 0.021954 | 0.020\* |  |
| C11 | 0.62896 (14) | 0.19495 (8) | −0.04491 (5) | 0.0198 (2) |  |
| H11 | 0.613347 | 0.148260 | −0.063292 | 0.024\* |  |
| C12 | 0.69673 (14) | 0.25342 (8) | −0.06849 (5) | 0.0213 (3) |  |
| H12 | 0.729058 | 0.246773 | −0.102839 | 0.026\* |  |
| C13 | 0.71752 (15) | 0.32195 (9) | −0.04192 (5) | 0.0225 (3) |  |
| H13 | 0.763208 | 0.362345 | −0.058308 | 0.027\* |  |
| C14 | 0.67164 (14) | 0.33170 (8) | 0.00870 (5) | 0.0188 (2) |  |
| H14 | 0.685451 | 0.378880 | 0.026511 | 0.023\* |  |
| C15 | 0.36613 (13) | 0.33712 (7) | 0.07575 (5) | 0.0145 (2) |  |
| C16 | 0.24015 (14) | 0.29442 (8) | 0.05757 (5) | 0.0187 (2) |  |
| H16 | 0.246233 | 0.241113 | 0.058428 | 0.022\* |  |
| C17 | 0.10591 (14) | 0.32883 (8) | 0.03823 (5) | 0.0223 (3) |  |
| H17 | 0.021498 | 0.299132 | 0.025636 | 0.027\* |  |
| C18 | 0.09603 (15) | 0.40663 (9) | 0.03743 (5) | 0.0244 (3) |  |
| H18 | 0.004221 | 0.430387 | 0.025019 | 0.029\* |  |
| C19 | 0.22024 (16) | 0.44958 (8) | 0.05477 (6) | 0.0251 (3) |  |
| H19 | 0.213155 | 0.502859 | 0.054087 | 0.030\* |  |
| C20 | 0.35557 (14) | 0.41551 (7) | 0.07322 (5) | 0.0194 (2) |  |
| H20 | 0.440703 | 0.445552 | 0.084079 | 0.023\* |  |
| C21 | 0.66857 (13) | 0.35214 (7) | 0.13420 (4) | 0.0137 (2) |  |
| C22 | 0.82075 (13) | 0.34249 (7) | 0.13237 (5) | 0.0169 (2) |  |
| H22 | 0.853714 | 0.305226 | 0.109456 | 0.020\* |  |
| C23 | 0.92369 (14) | 0.38739 (8) | 0.16407 (5) | 0.0199 (2) |  |
| H23 | 1.026643 | 0.380742 | 0.162608 | 0.024\* |  |
| C24 | 0.87633 (15) | 0.44197 (8) | 0.19791 (5) | 0.0208 (2) |  |
| H24 | 0.946840 | 0.472871 | 0.219150 | 0.025\* |  |
| C25 | 0.72615 (15) | 0.45121 (7) | 0.20057 (5) | 0.0193 (2) |  |
| H25 | 0.693907 | 0.488315 | 0.223766 | 0.023\* |  |
| C26 | 0.62226 (14) | 0.40605 (7) | 0.16923 (5) | 0.0162 (2) |  |
| H26 | 0.519633 | 0.411947 | 0.171685 | 0.019\* |  |
| C27 | 0.85540 (12) | 0.31824 (7) | 0.32212 (5) | 0.0135 (2) |  |
| C28 | 0.95012 (13) | 0.27317 (7) | 0.29597 (5) | 0.0169 (2) |  |
| H28 | 0.921031 | 0.223813 | 0.284488 | 0.020\* |  |
| C29 | 1.08716 (14) | 0.30144 (8) | 0.28702 (5) | 0.0200 (2) |  |
| H29 | 1.151400 | 0.271516 | 0.268846 | 0.024\* |  |
| C30 | 1.13055 (14) | 0.37306 (8) | 0.30446 (5) | 0.0210 (2) |  |
| H30 | 1.223740 | 0.392179 | 0.297667 | 0.025\* |  |
| C31 | 1.03898 (13) | 0.41695 (8) | 0.33172 (5) | 0.0191 (2) |  |
| H31 | 1.070734 | 0.465372 | 0.344517 | 0.023\* |  |
| C32 | 0.90055 (13) | 0.39010 (7) | 0.34036 (5) | 0.0154 (2) |  |
| H32 | 0.836944 | 0.420400 | 0.358547 | 0.018\* |  |
| C33 | 0.58470 (13) | 0.34724 (7) | 0.37171 (5) | 0.0162 (2) |  |
| C34 | 0.53274 (14) | 0.41623 (8) | 0.34941 (5) | 0.0214 (3) |  |
| H34 | 0.549894 | 0.429734 | 0.313930 | 0.026\* |  |
| C35 | 0.45616 (15) | 0.46475 (9) | 0.37934 (7) | 0.0306 (3) |  |
| H35 | 0.420563 | 0.511510 | 0.364434 | 0.037\* |  |
| C36 | 0.43204 (16) | 0.44459 (10) | 0.43098 (7) | 0.0351 (4) |  |
| H36 | 0.378966 | 0.477699 | 0.451270 | 0.042\* |  |
| C37 | 0.48378 (17) | 0.37715 (10) | 0.45355 (6) | 0.0325 (4) |  |
| H37 | 0.466493 | 0.364337 | 0.489145 | 0.039\* |  |
| C38 | 0.56163 (15) | 0.32762 (8) | 0.42411 (5) | 0.0233 (3) |  |
| H38 | 0.598222 | 0.281339 | 0.439562 | 0.028\* |  |
| C39 | 0.69439 (13) | 0.19343 (7) | 0.36416 (5) | 0.0155 (2) |  |
| C40 | 0.56826 (14) | 0.14868 (8) | 0.36508 (5) | 0.0186 (2) |  |
| H40 | 0.474281 | 0.165616 | 0.348174 | 0.022\* |  |
| C41 | 0.58142 (15) | 0.07944 (8) | 0.39086 (5) | 0.0217 (3) |  |
| H41 | 0.496283 | 0.048708 | 0.391445 | 0.026\* |  |
| C42 | 0.71826 (17) | 0.05501 (8) | 0.41571 (5) | 0.0246 (3) |  |
| H42 | 0.726633 | 0.007179 | 0.432788 | 0.029\* |  |
| C43 | 0.84305 (16) | 0.09954 (8) | 0.41593 (6) | 0.0258 (3) |  |
| H43 | 0.936155 | 0.082706 | 0.433713 | 0.031\* |  |
| C44 | 0.83189 (15) | 0.16903 (8) | 0.39007 (5) | 0.0211 (2) |  |
| H44 | 0.917303 | 0.199724 | 0.390029 | 0.025\* |  |
| C45 | 0.68939 (16) | 0.00183 (9) | 0.03779 (5) | 0.0250 (3) |  |
| H45A | 0.665296 | 0.035475 | 0.006737 | 0.038\* |  |
| H45B | 0.622336 | −0.041520 | 0.033938 | 0.038\* |  |
| H45C | 0.792058 | −0.015554 | 0.039308 | 0.038\* |  |
| F1 | 0.39357 (10) | 0.47983 (5) | 0.23666 (4) | 0.0312 (2) |  |
| F2 | 0.25298 (11) | 0.64373 (5) | 0.21053 (5) | 0.0403 (2) |  |
| P3 | 0.32448 (4) | 0.56186 (2) | 0.22344 (2) | 0.02096 (7) | 0.37 |
| F3 | 0.2106 (4) | 0.54962 (16) | 0.26530 (16) | 0.0271 (7)\* | 0.37 |
| F4 | 0.2034 (4) | 0.52417 (17) | 0.17613 (14) | 0.0287 (8)\* | 0.37 |
| F5 | 0.4294 (4) | 0.5771 (2) | 0.17871 (16) | 0.0364 (9)\* | 0.37 |
| F6 | 0.4418 (4) | 0.5997 (2) | 0.26859 (16) | 0.0354 (9)\* | 0.37 |
| P3A | 0.32448 (4) | 0.56186 (2) | 0.22344 (2) | 0.02096 (7) | 0.43 |
| F3A | 0.1811 (3) | 0.52250 (14) | 0.19014 (15) | 0.0259 (6)\* | 0.43 |
| F4A | 0.4034 (4) | 0.56212 (17) | 0.16926 (12) | 0.0268 (6)\* | 0.43 |
| F5A | 0.4666 (3) | 0.59915 (16) | 0.25598 (15) | 0.0280 (6)\* | 0.43 |
| F6A | 0.2465 (4) | 0.55559 (16) | 0.27815 (12) | 0.0266 (6)\* | 0.43 |
| P3B | 0.32448 (4) | 0.56186 (2) | 0.22344 (2) | 0.02096 (7) | 0.13 |
| F3B | 0.1640 (6) | 0.5296 (4) | 0.2111 (4) | 0.0215 (14)\* | 0.13 |
| F4B | 0.3488 (11) | 0.5597 (4) | 0.1627 (3) | 0.0243 (16)\* | 0.13 |
| F5B | 0.4940 (8) | 0.5955 (4) | 0.2370 (4) | 0.0305 (18)\* | 0.13 |
| F6B | 0.3016 (11) | 0.5704 (5) | 0.2882 (3) | 0.0288 (16)\* | 0.13 |
| P3C | 0.32448 (4) | 0.56186 (2) | 0.22344 (2) | 0.02096 (7) | 0.07 |
| F3C | 0.1707 (12) | 0.5445 (8) | 0.2403 (7) | 0.026 (3)\* | 0.07 |
| F4C | 0.268 (2) | 0.5397 (9) | 0.1629 (5) | 0.038 (3)\* | 0.07 |
| F5C | 0.4811 (14) | 0.5899 (9) | 0.2041 (7) | 0.036 (3)\* | 0.07 |
| F6C | 0.3860 (18) | 0.5892 (9) | 0.2862 (5) | 0.034 (3)\* | 0.07 |

# Atomic displacement parameters (Å2)

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | *U*11 | *U*22 | *U*33 | *U*12 | *U*13 | *U*23 |
| Fe1 | 0.01364 (8) | 0.01179 (8) | 0.00994 (7) | −0.00023 (6) | 0.00305 (5) | −0.00066 (6) |
| P1 | 0.01232 (12) | 0.01155 (13) | 0.01090 (12) | 0.00070 (10) | 0.00255 (9) | −0.00062 (10) |
| P2 | 0.01393 (13) | 0.01307 (13) | 0.01041 (12) | −0.00088 (10) | 0.00300 (9) | −0.00133 (10) |
| O1 | 0.0278 (5) | 0.0223 (5) | 0.0254 (5) | −0.0058 (4) | 0.0005 (4) | −0.0071 (4) |
| O2 | 0.0223 (5) | 0.0447 (7) | 0.0230 (5) | 0.0070 (4) | 0.0110 (4) | 0.0005 (5) |
| O3 | 0.0229 (4) | 0.0329 (6) | 0.0189 (4) | 0.0057 (4) | 0.0089 (3) | 0.0021 (4) |
| O4 | 0.0282 (5) | 0.0190 (4) | 0.0153 (4) | 0.0051 (4) | 0.0055 (3) | −0.0044 (3) |
| C1 | 0.0186 (5) | 0.0152 (5) | 0.0161 (5) | 0.0007 (4) | 0.0033 (4) | 0.0005 (4) |
| C2 | 0.0216 (6) | 0.0206 (6) | 0.0121 (5) | −0.0015 (5) | 0.0035 (4) | −0.0003 (4) |
| C3 | 0.0183 (5) | 0.0205 (6) | 0.0135 (5) | 0.0077 (4) | 0.0017 (4) | 0.0016 (4) |
| C4 | 0.0172 (5) | 0.0167 (5) | 0.0129 (5) | 0.0031 (4) | 0.0028 (4) | −0.0004 (4) |
| C5 | 0.0203 (5) | 0.0140 (5) | 0.0125 (5) | 0.0028 (4) | 0.0021 (4) | 0.0008 (4) |
| C6 | 0.0203 (5) | 0.0151 (5) | 0.0105 (5) | −0.0003 (4) | 0.0025 (4) | 0.0009 (4) |
| C7 | 0.0165 (5) | 0.0153 (5) | 0.0105 (4) | −0.0005 (4) | 0.0024 (4) | −0.0004 (4) |
| C8 | 0.0172 (5) | 0.0169 (5) | 0.0107 (5) | −0.0005 (4) | 0.0019 (4) | −0.0011 (4) |
| C9 | 0.0134 (5) | 0.0167 (5) | 0.0116 (5) | 0.0024 (4) | 0.0017 (4) | 0.0000 (4) |
| C10 | 0.0192 (5) | 0.0164 (5) | 0.0136 (5) | 0.0022 (4) | 0.0017 (4) | −0.0010 (4) |
| C11 | 0.0217 (6) | 0.0235 (6) | 0.0139 (5) | 0.0047 (5) | 0.0013 (4) | −0.0040 (5) |
| C12 | 0.0190 (6) | 0.0329 (7) | 0.0124 (5) | 0.0052 (5) | 0.0033 (4) | −0.0008 (5) |
| C13 | 0.0229 (6) | 0.0291 (7) | 0.0166 (5) | −0.0029 (5) | 0.0067 (4) | 0.0028 (5) |
| C14 | 0.0219 (6) | 0.0194 (6) | 0.0160 (5) | −0.0024 (5) | 0.0050 (4) | −0.0001 (4) |
| C15 | 0.0157 (5) | 0.0163 (5) | 0.0120 (5) | 0.0036 (4) | 0.0035 (4) | 0.0000 (4) |
| C16 | 0.0174 (5) | 0.0198 (6) | 0.0187 (5) | 0.0020 (4) | 0.0015 (4) | −0.0010 (5) |
| C17 | 0.0163 (5) | 0.0308 (7) | 0.0195 (6) | 0.0035 (5) | 0.0014 (4) | −0.0004 (5) |
| C18 | 0.0210 (6) | 0.0317 (7) | 0.0209 (6) | 0.0129 (5) | 0.0038 (5) | 0.0025 (5) |
| C19 | 0.0301 (7) | 0.0205 (6) | 0.0246 (6) | 0.0106 (5) | 0.0035 (5) | 0.0026 (5) |
| C20 | 0.0220 (6) | 0.0168 (6) | 0.0196 (5) | 0.0025 (5) | 0.0032 (4) | 0.0012 (5) |
| C21 | 0.0164 (5) | 0.0131 (5) | 0.0118 (5) | −0.0014 (4) | 0.0022 (4) | 0.0011 (4) |
| C22 | 0.0177 (5) | 0.0170 (5) | 0.0163 (5) | 0.0007 (4) | 0.0034 (4) | 0.0022 (4) |
| C23 | 0.0167 (5) | 0.0230 (6) | 0.0196 (6) | −0.0030 (5) | 0.0006 (4) | 0.0043 (5) |
| C24 | 0.0248 (6) | 0.0192 (6) | 0.0167 (5) | −0.0065 (5) | −0.0029 (4) | 0.0024 (5) |
| C25 | 0.0280 (6) | 0.0157 (6) | 0.0140 (5) | −0.0031 (5) | 0.0025 (4) | −0.0016 (4) |
| C26 | 0.0197 (5) | 0.0153 (5) | 0.0141 (5) | −0.0007 (4) | 0.0037 (4) | −0.0006 (4) |
| C27 | 0.0138 (5) | 0.0145 (5) | 0.0126 (5) | 0.0002 (4) | 0.0029 (4) | 0.0003 (4) |
| C28 | 0.0184 (5) | 0.0173 (6) | 0.0152 (5) | 0.0019 (4) | 0.0031 (4) | −0.0018 (4) |
| C29 | 0.0167 (5) | 0.0267 (7) | 0.0176 (5) | 0.0046 (5) | 0.0058 (4) | 0.0017 (5) |
| C30 | 0.0143 (5) | 0.0273 (7) | 0.0214 (6) | −0.0014 (5) | 0.0026 (4) | 0.0054 (5) |
| C31 | 0.0170 (5) | 0.0183 (6) | 0.0212 (6) | −0.0029 (4) | −0.0003 (4) | 0.0020 (5) |
| C32 | 0.0163 (5) | 0.0145 (5) | 0.0154 (5) | 0.0005 (4) | 0.0023 (4) | −0.0001 (4) |
| C33 | 0.0139 (5) | 0.0196 (6) | 0.0158 (5) | −0.0024 (4) | 0.0044 (4) | −0.0059 (4) |
| C34 | 0.0179 (5) | 0.0241 (7) | 0.0208 (6) | 0.0042 (5) | −0.0024 (4) | −0.0074 (5) |
| C35 | 0.0180 (6) | 0.0317 (8) | 0.0400 (8) | 0.0076 (5) | −0.0044 (5) | −0.0180 (7) |
| C36 | 0.0181 (6) | 0.0430 (9) | 0.0462 (9) | −0.0043 (6) | 0.0117 (6) | −0.0294 (8) |
| C37 | 0.0319 (7) | 0.0406 (9) | 0.0296 (7) | −0.0169 (7) | 0.0209 (6) | −0.0185 (7) |
| C38 | 0.0276 (7) | 0.0249 (7) | 0.0202 (6) | −0.0103 (5) | 0.0127 (5) | −0.0074 (5) |
| C39 | 0.0212 (5) | 0.0138 (5) | 0.0115 (5) | −0.0025 (4) | 0.0025 (4) | −0.0011 (4) |
| C40 | 0.0212 (6) | 0.0205 (6) | 0.0149 (5) | −0.0040 (5) | 0.0049 (4) | −0.0002 (4) |
| C41 | 0.0297 (6) | 0.0192 (6) | 0.0175 (5) | −0.0080 (5) | 0.0077 (5) | −0.0011 (5) |
| C42 | 0.0383 (7) | 0.0154 (6) | 0.0203 (6) | −0.0021 (5) | 0.0054 (5) | 0.0023 (5) |
| C43 | 0.0295 (7) | 0.0198 (6) | 0.0264 (7) | 0.0012 (5) | −0.0024 (5) | 0.0056 (5) |
| C44 | 0.0222 (6) | 0.0176 (6) | 0.0223 (6) | −0.0031 (5) | −0.0013 (5) | 0.0021 (5) |
| C45 | 0.0307 (7) | 0.0279 (7) | 0.0161 (5) | 0.0105 (6) | 0.0018 (5) | −0.0071 (5) |
| F1 | 0.0343 (5) | 0.0298 (5) | 0.0312 (5) | 0.0138 (4) | 0.0105 (4) | 0.0049 (4) |
| F2 | 0.0383 (5) | 0.0219 (5) | 0.0584 (7) | 0.0049 (4) | −0.0022 (5) | 0.0023 (4) |
| P3 | 0.01575 (14) | 0.02113 (17) | 0.02609 (17) | 0.00089 (12) | 0.00322 (12) | 0.00016 (13) |
| P3A | 0.01575 (14) | 0.02113 (17) | 0.02609 (17) | 0.00089 (12) | 0.00322 (12) | 0.00016 (13) |
| P3B | 0.01575 (14) | 0.02113 (17) | 0.02609 (17) | 0.00089 (12) | 0.00322 (12) | 0.00016 (13) |
| P3C | 0.01575 (14) | 0.02113 (17) | 0.02609 (17) | 0.00089 (12) | 0.00322 (12) | 0.00016 (13) |

# Geometric parameters (Å, º)

|  |  |  |  |
| --- | --- | --- | --- |
| Fe1—P1 | 2.2334 (3) | C26—H26 | 0.9500 |
| Fe1—C1 | 1.7769 (12) | C27—C28 | 1.4013 (16) |
| Fe1—C2 | 1.7762 (13) | C27—C32 | 1.3998 (17) |
| Fe1—C4 | 2.1312 (12) | C28—H28 | 0.9500 |
| Fe1—C5 | 2.0497 (12) | C28—C29 | 1.3906 (18) |
| Fe1—C6 | 2.0717 (11) | C29—H29 | 0.9500 |
| Fe1—C7 | 2.1431 (11) | C29—C30 | 1.386 (2) |
| P1—C9 | 1.8379 (11) | C30—H30 | 0.9500 |
| P1—C15 | 1.8394 (12) | C30—C31 | 1.3857 (19) |
| P1—C21 | 1.8212 (12) | C31—H31 | 0.9500 |
| P2—C8 | 1.8298 (12) | C31—C32 | 1.3911 (17) |
| P2—C27 | 1.7929 (12) | C32—H32 | 0.9500 |
| P2—C33 | 1.7939 (12) | C33—C34 | 1.4030 (19) |
| P2—C39 | 1.7936 (13) | C33—C38 | 1.3959 (18) |
| O1—C1 | 1.1545 (15) | C34—H34 | 0.9500 |
| O2—C2 | 1.1428 (16) | C34—C35 | 1.3889 (19) |
| O3—C3 | 1.2071 (15) | C35—H35 | 0.9500 |
| O4—C3 | 1.3621 (16) | C35—C36 | 1.383 (3) |
| O4—C45 | 1.4382 (15) | C36—H36 | 0.9500 |
| C3—C4 | 1.4719 (17) | C36—C37 | 1.381 (3) |
| C4—H4 | 0.966 (16) | C37—H37 | 0.9500 |
| C4—C5 | 1.4312 (17) | C37—C38 | 1.399 (2) |
| C5—H5 | 0.969 (17) | C38—H38 | 0.9500 |
| C5—C6 | 1.4052 (16) | C39—C40 | 1.3996 (17) |
| C6—H6 | 0.982 (16) | C39—C44 | 1.3974 (18) |
| C6—C7 | 1.4260 (17) | C40—H40 | 0.9500 |
| C7—H7 | 0.987 (16) | C40—C41 | 1.3871 (19) |
| C7—C8 | 1.5097 (16) | C41—H41 | 0.9500 |
| C8—H8A | 0.9900 | C41—C42 | 1.384 (2) |
| C8—H8B | 0.9900 | C42—H42 | 0.9500 |
| C9—C10 | 1.3928 (17) | C42—C43 | 1.384 (2) |
| C9—C14 | 1.4005 (17) | C43—H43 | 0.9500 |
| C10—H10 | 0.9500 | C43—C44 | 1.3917 (19) |
| C10—C11 | 1.3979 (17) | C44—H44 | 0.9500 |
| C11—H11 | 0.9500 | C45—H45A | 0.9800 |
| C11—C12 | 1.381 (2) | C45—H45B | 0.9800 |
| C12—H12 | 0.9500 | C45—H45C | 0.9800 |
| C12—C13 | 1.388 (2) | F1—P3 | 1.6054 (10) |
| C13—H13 | 0.9500 | F1—P3A | 1.6054 (10) |
| C13—C14 | 1.3932 (17) | F1—P3B | 1.6054 (10) |
| C14—H14 | 0.9500 | F1—P3C | 1.6054 (10) |
| C15—C16 | 1.3987 (17) | F2—P3 | 1.6099 (10) |
| C15—C20 | 1.3987 (18) | F2—P3A | 1.6099 (10) |
| C16—H16 | 0.9500 | F2—P3B | 1.6099 (10) |
| C16—C17 | 1.3929 (18) | F2—P3C | 1.6099 (10) |
| C17—H17 | 0.9500 | P3—F3 | 1.583 (3) |
| C17—C18 | 1.387 (2) | P3—F4 | 1.644 (3) |
| C18—H18 | 0.9500 | P3—F5 | 1.587 (3) |
| C18—C19 | 1.384 (2) | P3—F6 | 1.593 (3) |
| C19—H19 | 0.9500 | P3A—F3A | 1.609 (2) |
| C19—C20 | 1.3937 (18) | P3A—F4A | 1.614 (3) |
| C20—H20 | 0.9500 | P3A—F5A | 1.577 (3) |
| C21—C22 | 1.4023 (16) | P3A—F6A | 1.625 (2) |
| C21—C26 | 1.4003 (17) | P3B—F3B | 1.560 (6) |
| C22—H22 | 0.9500 | P3B—F4B | 1.562 (6) |
| C22—C23 | 1.3929 (18) | P3B—F5B | 1.647 (7) |
| C23—H23 | 0.9500 | P3B—F6B | 1.664 (6) |
| C23—C24 | 1.393 (2) | P3C—F3C | 1.546 (9) |
| C24—H24 | 0.9500 | P3C—F4C | 1.576 (10) |
| C24—C25 | 1.3871 (19) | P3C—F5C | 1.643 (10) |
| C25—H25 | 0.9500 | P3C—F6C | 1.663 (10) |
| C25—C26 | 1.3964 (17) |  |  |
|  |  |  |  |
| C1—Fe1—P1 | 102.27 (4) | C25—C26—H26 | 119.8 |
| C1—Fe1—C4 | 97.93 (5) | C28—C27—P2 | 119.38 (9) |
| C1—Fe1—C5 | 94.19 (5) | C32—C27—P2 | 120.49 (9) |
| C1—Fe1—C6 | 120.19 (5) | C32—C27—C28 | 120.12 (11) |
| C1—Fe1—C7 | 159.30 (5) | C27—C28—H28 | 120.4 |
| C2—Fe1—P1 | 95.53 (4) | C29—C28—C27 | 119.30 (12) |
| C2—Fe1—C1 | 88.28 (6) | C29—C28—H28 | 120.4 |
| C2—Fe1—C4 | 167.12 (5) | C28—C29—H29 | 119.8 |
| C2—Fe1—C5 | 128.65 (5) | C30—C29—C28 | 120.38 (12) |
| C2—Fe1—C6 | 96.57 (5) | C30—C29—H29 | 119.8 |
| C2—Fe1—C7 | 90.50 (5) | C29—C30—H30 | 119.8 |
| C4—Fe1—P1 | 94.16 (3) | C31—C30—C29 | 120.47 (12) |
| C4—Fe1—C7 | 79.70 (4) | C31—C30—H30 | 119.8 |
| C5—Fe1—P1 | 133.35 (4) | C30—C31—H31 | 120.0 |
| C5—Fe1—C4 | 39.98 (5) | C30—C31—C32 | 120.00 (12) |
| C5—Fe1—C6 | 39.87 (5) | C32—C31—H31 | 120.0 |
| C5—Fe1—C7 | 70.66 (5) | C27—C32—H32 | 120.2 |
| C6—Fe1—P1 | 136.02 (4) | C31—C32—C27 | 119.69 (11) |
| C6—Fe1—C4 | 70.55 (5) | C31—C32—H32 | 120.2 |
| C6—Fe1—C7 | 39.51 (5) | C34—C33—P2 | 119.26 (9) |
| C7—Fe1—P1 | 98.42 (3) | C38—C33—P2 | 120.41 (11) |
| C9—P1—Fe1 | 119.63 (4) | C38—C33—C34 | 120.30 (12) |
| C9—P1—C15 | 98.79 (5) | C33—C34—H34 | 120.1 |
| C15—P1—Fe1 | 115.04 (4) | C35—C34—C33 | 119.82 (14) |
| C21—P1—Fe1 | 111.13 (4) | C35—C34—H34 | 120.1 |
| C21—P1—C9 | 103.52 (5) | C34—C35—H35 | 120.2 |
| C21—P1—C15 | 107.24 (6) | C36—C35—C34 | 119.60 (15) |
| C27—P2—C8 | 110.34 (5) | C36—C35—H35 | 120.2 |
| C27—P2—C33 | 110.54 (6) | C35—C36—H36 | 119.4 |
| C27—P2—C39 | 109.64 (6) | C37—C36—C35 | 121.12 (13) |
| C33—P2—C8 | 106.77 (6) | C37—C36—H36 | 119.4 |
| C39—P2—C8 | 110.13 (6) | C36—C37—H37 | 119.9 |
| C39—P2—C33 | 109.37 (6) | C36—C37—C38 | 120.14 (14) |
| C3—O4—C45 | 116.27 (11) | C38—C37—H37 | 119.9 |
| O1—C1—Fe1 | 175.30 (11) | C33—C38—C37 | 119.01 (15) |
| O2—C2—Fe1 | 177.73 (12) | C33—C38—H38 | 120.5 |
| O3—C3—O4 | 122.80 (12) | C37—C38—H38 | 120.5 |
| O3—C3—C4 | 125.21 (13) | C40—C39—P2 | 119.17 (10) |
| O4—C3—C4 | 111.99 (10) | C44—C39—P2 | 120.74 (9) |
| Fe1—C4—H4 | 109.7 (10) | C44—C39—C40 | 120.06 (12) |
| C3—C4—Fe1 | 120.97 (8) | C39—C40—H40 | 120.2 |
| C3—C4—H4 | 111.5 (10) | C41—C40—C39 | 119.55 (12) |
| C5—C4—Fe1 | 66.94 (7) | C41—C40—H40 | 120.2 |
| C5—C4—C3 | 119.94 (11) | C40—C41—H41 | 119.9 |
| C5—C4—H4 | 120.6 (10) | C42—C41—C40 | 120.16 (12) |
| Fe1—C5—H5 | 124.0 (9) | C42—C41—H41 | 119.9 |
| C4—C5—Fe1 | 73.08 (7) | C41—C42—H42 | 119.7 |
| C4—C5—H5 | 120.2 (10) | C41—C42—C43 | 120.68 (13) |
| C6—C5—Fe1 | 70.91 (7) | C43—C42—H42 | 119.7 |
| C6—C5—C4 | 117.72 (11) | C42—C43—H43 | 120.1 |
| C6—C5—H5 | 122.0 (10) | C42—C43—C44 | 119.88 (13) |
| Fe1—C6—H6 | 121.7 (9) | C44—C43—H43 | 120.1 |
| C5—C6—Fe1 | 69.22 (7) | C39—C44—H44 | 120.2 |
| C5—C6—H6 | 120.3 (10) | C43—C44—C39 | 119.65 (12) |
| C5—C6—C7 | 117.91 (11) | C43—C44—H44 | 120.2 |
| C7—C6—Fe1 | 72.95 (7) | O4—C45—H45A | 109.5 |
| C7—C6—H6 | 121.2 (10) | O4—C45—H45B | 109.5 |
| Fe1—C7—H7 | 104.3 (9) | O4—C45—H45C | 109.5 |
| C6—C7—Fe1 | 67.55 (6) | H45A—C45—H45B | 109.5 |
| C6—C7—H7 | 120.7 (10) | H45A—C45—H45C | 109.5 |
| C6—C7—C8 | 118.60 (10) | H45B—C45—H45C | 109.5 |
| C8—C7—Fe1 | 125.88 (8) | F1—P3—F2 | 179.14 (6) |
| C8—C7—H7 | 112.7 (10) | F1—P3—F4 | 89.00 (11) |
| P2—C8—H8A | 108.9 | F2—P3—F4 | 90.69 (11) |
| P2—C8—H8B | 108.9 | F3—P3—F1 | 90.72 (11) |
| C7—C8—P2 | 113.49 (8) | F3—P3—F2 | 88.47 (12) |
| C7—C8—H8A | 108.9 | F3—P3—F4 | 88.98 (19) |
| C7—C8—H8B | 108.9 | F3—P3—F5 | 175.89 (19) |
| H8A—C8—H8B | 107.7 | F3—P3—F6 | 91.6 (2) |
| C10—C9—P1 | 120.27 (9) | F5—P3—F1 | 92.47 (14) |
| C10—C9—C14 | 118.61 (11) | F5—P3—F2 | 88.32 (14) |
| C14—C9—P1 | 121.02 (9) | F5—P3—F4 | 88.5 (2) |
| C9—C10—H10 | 119.6 | F5—P3—F6 | 90.9 (2) |
| C9—C10—C11 | 120.73 (12) | F6—P3—F1 | 91.86 (13) |
| C11—C10—H10 | 119.6 | F6—P3—F2 | 88.46 (13) |
| C10—C11—H11 | 120.0 | F6—P3—F4 | 178.96 (19) |
| C12—C11—C10 | 120.10 (12) | F1—P3A—F2 | 179.14 (6) |
| C12—C11—H11 | 120.0 | F1—P3A—F3A | 88.75 (10) |
| C11—C12—H12 | 120.1 | F1—P3A—F4A | 88.19 (11) |
| C11—C12—C13 | 119.87 (12) | F1—P3A—F6A | 88.20 (10) |
| C13—C12—H12 | 120.1 | F2—P3A—F4A | 92.49 (11) |
| C12—C13—H13 | 119.9 | F2—P3A—F6A | 91.11 (10) |
| C12—C13—C14 | 120.25 (12) | F3A—P3A—F2 | 90.73 (10) |
| C14—C13—H13 | 119.9 | F3A—P3A—F4A | 89.47 (19) |
| C9—C14—H14 | 119.8 | F3A—P3A—F6A | 89.29 (18) |
| C13—C14—C9 | 120.43 (12) | F4A—P3A—F6A | 176.20 (14) |
| C13—C14—H14 | 119.8 | F5A—P3A—F1 | 90.32 (11) |
| C16—C15—P1 | 117.00 (9) | F5A—P3A—F2 | 90.20 (11) |
| C16—C15—C20 | 118.59 (11) | F5A—P3A—F3A | 179.02 (16) |
| C20—C15—P1 | 124.37 (9) | F5A—P3A—F4A | 90.2 (2) |
| C15—C16—H16 | 119.5 | F5A—P3A—F6A | 91.00 (19) |
| C17—C16—C15 | 121.04 (13) | F1—P3B—F2 | 179.14 (6) |
| C17—C16—H16 | 119.5 | F1—P3B—F5B | 87.6 (3) |
| C16—C17—H17 | 120.1 | F1—P3B—F6B | 88.9 (3) |
| C18—C17—C16 | 119.71 (13) | F2—P3B—F5B | 93.1 (3) |
| C18—C17—H17 | 120.1 | F2—P3B—F6B | 90.6 (3) |
| C17—C18—H18 | 120.1 | F3B—P3B—F1 | 92.1 (3) |
| C19—C18—C17 | 119.85 (12) | F3B—P3B—F2 | 87.2 (3) |
| C19—C18—H18 | 120.1 | F3B—P3B—F4B | 93.0 (5) |
| C18—C19—H19 | 119.6 | F3B—P3B—F5B | 179.5 (5) |
| C18—C19—C20 | 120.72 (13) | F3B—P3B—F6B | 89.3 (5) |
| C20—C19—H19 | 119.6 | F4B—P3B—F1 | 94.3 (3) |
| C15—C20—H20 | 120.0 | F4B—P3B—F2 | 86.2 (3) |
| C19—C20—C15 | 120.05 (12) | F4B—P3B—F5B | 87.5 (5) |
| C19—C20—H20 | 120.0 | F4B—P3B—F6B | 176.0 (4) |
| C22—C21—P1 | 119.24 (9) | F5B—P3B—F6B | 90.3 (5) |
| C26—C21—P1 | 121.00 (9) | F1—P3C—F2 | 179.14 (6) |
| C26—C21—C22 | 119.09 (11) | F1—P3C—F5C | 90.3 (5) |
| C21—C22—H22 | 119.9 | F1—P3C—F6C | 90.0 (5) |
| C23—C22—C21 | 120.17 (12) | F2—P3C—F5C | 90.6 (5) |
| C23—C22—H22 | 119.9 | F2—P3C—F6C | 89.9 (5) |
| C22—C23—H23 | 119.9 | F3C—P3C—F1 | 96.1 (5) |
| C24—C23—C22 | 120.28 (12) | F3C—P3C—F2 | 83.0 (5) |
| C24—C23—H23 | 119.9 | F3C—P3C—F4C | 91.3 (8) |
| C23—C24—H24 | 120.0 | F3C—P3C—F5C | 173.6 (7) |
| C25—C24—C23 | 119.96 (12) | F3C—P3C—F6C | 90.3 (8) |
| C25—C24—H24 | 120.0 | F4C—P3C—F1 | 92.1 (6) |
| C24—C25—H25 | 119.9 | F4C—P3C—F2 | 88.0 (6) |
| C24—C25—C26 | 120.13 (12) | F4C—P3C—F5C | 88.7 (9) |
| C26—C25—H25 | 119.9 | F4C—P3C—F6C | 177.2 (9) |
| C21—C26—H26 | 119.8 | F5C—P3C—F6C | 89.6 (8) |
| C25—C26—C21 | 120.34 (11) |  |  |
|  |  |  |  |
| Fe1—P1—C9—C10 | 25.81 (11) | C15—P1—C21—C26 | 41.49 (11) |
| Fe1—P1—C9—C14 | −158.01 (8) | C15—C16—C17—C18 | −0.7 (2) |
| Fe1—P1—C15—C16 | −43.04 (10) | C16—C15—C20—C19 | 2.36 (18) |
| Fe1—P1—C15—C20 | 139.46 (9) | C16—C17—C18—C19 | 1.4 (2) |
| Fe1—P1—C21—C22 | 85.53 (10) | C17—C18—C19—C20 | −0.2 (2) |
| Fe1—P1—C21—C26 | −85.02 (10) | C18—C19—C20—C15 | −1.7 (2) |
| Fe1—C4—C5—C6 | −56.90 (9) | C20—C15—C16—C17 | −1.17 (18) |
| Fe1—C5—C6—C7 | −56.23 (10) | C21—P1—C9—C10 | 150.09 (9) |
| Fe1—C6—C7—C8 | 119.80 (10) | C21—P1—C9—C14 | −33.73 (11) |
| Fe1—C7—C8—P2 | 167.38 (6) | C21—P1—C15—C16 | −167.19 (9) |
| P1—C9—C10—C11 | 177.44 (9) | C21—P1—C15—C20 | 15.30 (12) |
| P1—C9—C14—C13 | −177.68 (10) | C21—C22—C23—C24 | 0.21 (19) |
| P1—C15—C16—C17 | −178.82 (10) | C22—C21—C26—C25 | 2.07 (18) |
| P1—C15—C20—C19 | 179.83 (10) | C22—C23—C24—C25 | 0.73 (19) |
| P1—C21—C22—C23 | −172.32 (9) | C23—C24—C25—C26 | −0.25 (19) |
| P1—C21—C26—C25 | 172.62 (9) | C24—C25—C26—C21 | −1.16 (19) |
| P2—C27—C28—C29 | 177.59 (9) | C26—C21—C22—C23 | −1.59 (18) |
| P2—C27—C32—C31 | −178.52 (9) | C27—P2—C8—C7 | 55.85 (10) |
| P2—C33—C34—C35 | 177.10 (10) | C27—P2—C33—C34 | 71.33 (11) |
| P2—C33—C38—C37 | −176.86 (10) | C27—P2—C33—C38 | −110.66 (11) |
| P2—C39—C40—C41 | −179.33 (9) | C27—P2—C39—C40 | −166.48 (9) |
| P2—C39—C44—C43 | 179.03 (11) | C27—P2—C39—C44 | 15.56 (12) |
| O3—C3—C4—Fe1 | 121.25 (12) | C27—C28—C29—C30 | 0.92 (19) |
| O3—C3—C4—C5 | −159.20 (12) | C28—C27—C32—C31 | 0.91 (17) |
| O4—C3—C4—Fe1 | −57.61 (13) | C28—C29—C30—C31 | 0.95 (19) |
| O4—C3—C4—C5 | 21.94 (15) | C29—C30—C31—C32 | −1.91 (19) |
| C3—C4—C5—Fe1 | −113.59 (10) | C30—C31—C32—C27 | 0.97 (18) |
| C3—C4—C5—C6 | −170.50 (11) | C32—C27—C28—C29 | −1.85 (18) |
| C4—C5—C6—Fe1 | 58.01 (9) | C33—P2—C8—C7 | 176.03 (9) |
| C4—C5—C6—C7 | 1.78 (16) | C33—P2—C27—C28 | 174.08 (9) |
| C5—C6—C7—Fe1 | 54.38 (9) | C33—P2—C27—C32 | −6.48 (11) |
| C5—C6—C7—C8 | 174.18 (10) | C33—P2—C39—C40 | 72.15 (11) |
| C6—C7—C8—P2 | 85.57 (12) | C33—P2—C39—C44 | −105.80 (11) |
| C8—P2—C27—C28 | −68.04 (11) | C33—C34—C35—C36 | 0.1 (2) |
| C8—P2—C27—C32 | 111.40 (10) | C34—C33—C38—C37 | 1.13 (19) |
| C8—P2—C33—C34 | −48.72 (11) | C34—C35—C36—C37 | 0.5 (2) |
| C8—P2—C33—C38 | 129.30 (10) | C35—C36—C37—C38 | −0.3 (2) |
| C8—P2—C39—C40 | −44.89 (11) | C36—C37—C38—C33 | −0.5 (2) |
| C8—P2—C39—C44 | 137.16 (10) | C38—C33—C34—C35 | −0.92 (19) |
| C9—P1—C15—C16 | 85.61 (10) | C39—P2—C8—C7 | −65.33 (10) |
| C9—P1—C15—C20 | −91.89 (11) | C39—P2—C27—C28 | 53.43 (11) |
| C9—P1—C21—C22 | −44.12 (11) | C39—P2—C27—C32 | −127.14 (10) |
| C9—P1—C21—C26 | 145.34 (10) | C39—P2—C33—C34 | −167.85 (10) |
| C9—C10—C11—C12 | 0.00 (18) | C39—P2—C33—C38 | 10.16 (12) |
| C10—C9—C14—C13 | −1.45 (18) | C39—C40—C41—C42 | 0.33 (19) |
| C10—C11—C12—C13 | −0.93 (19) | C40—C39—C44—C43 | 1.10 (19) |
| C11—C12—C13—C14 | 0.7 (2) | C40—C41—C42—C43 | 1.0 (2) |
| C12—C13—C14—C9 | 0.5 (2) | C41—C42—C43—C44 | −1.2 (2) |
| C14—C9—C10—C11 | 1.18 (18) | C42—C43—C44—C39 | 0.2 (2) |
| C15—P1—C9—C10 | −99.69 (10) | C44—C39—C40—C41 | −1.36 (18) |
| C15—P1—C9—C14 | 76.49 (11) | C45—O4—C3—O3 | −3.63 (17) |
| C15—P1—C21—C22 | −147.96 (9) | C45—O4—C3—C4 | 175.26 (10) |

# Hydrogen-bond geometry (Å, º)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
| C26—H26···F1 | 0.95 | 2.43 | 3.1353 (15) | 131 |
| C28—H28···F5*B*i | 0.95 | 2.49 | 3.322 (8) | 146 |
| C28—H28···F5*C*i | 0.95 | 2.55 | 3.320 (15) | 139 |
| C32—H32···O4ii | 0.95 | 2.56 | 3.3659 (16) | 143 |
| C34—H34···F1 | 0.95 | 2.41 | 3.1349 (16) | 133 |
| C40—H40···F2iii | 0.95 | 2.40 | 3.2586 (16) | 150 |

|  |
| --- |
| Symmetry codes: (i) −*x*+3/2, *y*−1/2, −*z*+1/2; (ii) −*x*+3/2, *y*+1/2, −*z*+1/2; (iii) −*x*+1/2, *y*−1/2, −*z*+1/2. |

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