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1,4-Bis{5-[bis(propan-2-yloxy)phosphoryl]thiophen-2-yl}-2,5-dipropoxybenzene

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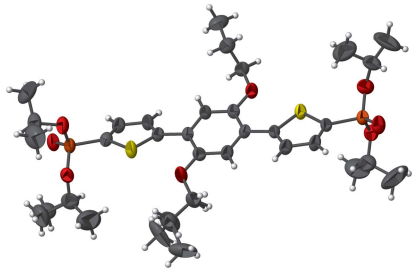
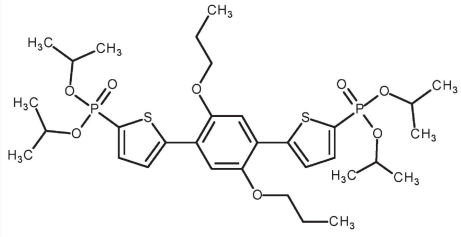
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{32}H_{48}O_8P_2S_2$, was prepared by the nickel-catalyzed reaction of bis(5-bromothienyl)dipropoxybenzene and triisopropyl phosphite. The thiophene rings are inclined to the benzene ring by 14.6 (2) and 25.3 (3)°. One propyloxy group is disordered. Four hydrogen bonds connect the molecules, which are arranged in ribbons parallel to the *bc* plane.

3D view



Chemical scheme



Structure description

The title compound, Fig. 1, was prepared by nickel-catalyzed reaction of bis-5-bromothienyl-dipropoxybenzene and triisopropyl phosphite.

Bisphosphonates are used to treat osteoporosis (Winckler *et al.*, 1996; Pavlov *et al.*, 2016). For layered materials from bisphosphonates, see: Ansell *et al.* (1997); Lecollinet *et al.* (2009). Bisphosphonates can act also as bifunctional building blocks for stilbenoid oligomers (Detert & Sugiono, 2000).

A phase transition of the crystals appears on cooling to 248 K. After this, the crystals were not suitable for single-crystal measurements. Thus, the data collection was performed at 248 K. There is one non-symmetric molecule in the asymmetric unit of the monoclinic unit cell. The dihedral angles between the mean planes of the essentially planar thiophene rings and the central benzene ring are 25.3 (2) and 14.6 (2)°.

In the crystal, four C—H···O hydrogen bonds connect one molecule with three symmetry-related ones (see Table 1) and form undulating sheets (Fig. 2) parallel to the *bc* plane.

Synthesis and crystallization

A mixture composed of 1,4-bis-5-bromothienyl-2,5-dipropoxybenzene (Theisinger, 2005) (1.1 g, 2.13 mmol), anhydrous nickel chloride (0.1 g, 0.78 mmol) and triisopropyl phos-

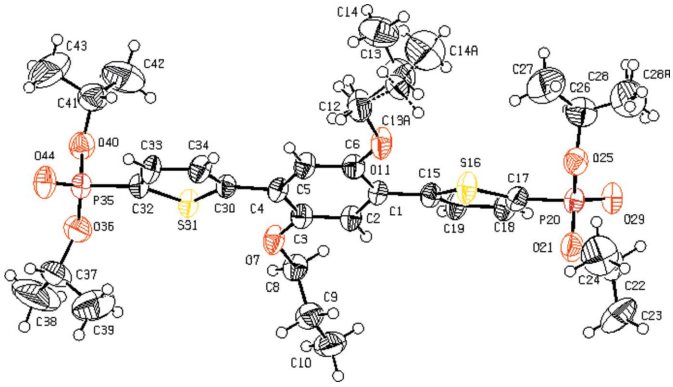


Figure 1
A view of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Disordered groups are shown with dashed lines.

phite (0.92 g, 4.4 mmol) was stirred for 3 h at 450 K under nitrogen. After 2 h, an additional portion of $NiCl_2$ was added. After completion of the reaction, excess phosphite was removed *in vacuo* and the product purified *via* column chromatography (silica gel, petroleum ether:ethyl acetate = 1:1) to afford 1.01 g of the title compound in 70% yield. IR: 3066, 2978, 2935, 1531, 1439, 1387, 1336, 1252, 1217, 1178, 1142, 1039, 985, 885, 823, 775, 669, 588. ^{1}H NMR ($CDCl_3$) δ: 7.60 (*dd*, 2 H, J_{HP} = 8.5 Hz, J_{HH} = 4.1 Hz), 7.55 (*t*, 2 H, $J_{HH} = J_{HP}$ = 3.7 Hz), 7.26 (*s*, 2 H), 4.7 (*m*, 4 H), 4.06 (*t*, 4 H, OCH_2), 1.19 (*m*, 4 H), 1.32 (*dd*, 24 H), 1.09 (*t*, 6 H). ^{13}C NMR ($CDCl_3$) δ: 149.4, 146.6 ($^4J_{CP}$ = 7.9 Hz), 136.0 (J_{CP} = 11.2 Hz), 129.1 (J_{CP} = 207.8 Hz), 125.8 (J_{CP} = 16.9 Hz), 122.8, 112.4, 71.4, 71.3, 24.0 (J_{CP} = 4.5), 22.7, 10.9. FD–MS: 1372.6 (4%, M_2^+), 686.2 (100%, M^+). Yellow crystals with m.p. 458 K were obtained by recrystallization from a dichloromethane solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. One *n*-propyloxy group is disordered over two positions with a site occupation factor of 0.75 (2) for the major occupied site. The C—C distances involving the disordered atoms were restrained to 1.54 (2) Å. In addition, one methyl group of an isopropyl group is disordered over two equally occupied positions. For the atoms C12, C13*A*, C14*A*, C26, C27 and C28*A*, a rigid-bond restraint (RIGU) was applied.

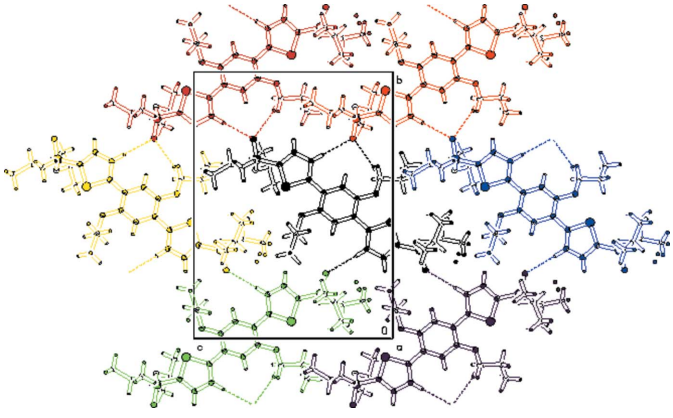


Figure 2
A partial packing diagram. Hydrogen bonds are shown as dashed lines. The view is along the *a* axis.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O29^{i}	0.98	2.61	3.533 (9)	156
C12—H12*C*···O44^{ii}	0.98	2.52	3.480 (10)	166
C19—H19···O29^{i}	0.94	2.51	3.436 (8)	169
C34—H34···O44^{ii}	0.94	2.49	3.424 (8)	174

Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{32}H_{48}O_8P_2S_2$
M_r	686.76
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	248
a, *b*, *c* (Å)	13.6507 (8), 18.8828 (11), 14.5033 (8)
β (°)	103.279 (5)
V (Å^3)	3638.5 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.28
Crystal size (mm)	0.40 × 0.15 × 0.08
Data collection	
Diffractometer	Stoe IPDS 2T
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	25084, 8750, 3346
R_{int}	0.143
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.661
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.091, 0.304, 0.95
No. of reflections	8750
No. of parameters	433
No. of restraints	22
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.73, −0.53

Computer programs: *X-AREA* and *X-RED* (Stoe & Cie, 1996), *SIR97* (Altomare *et al.*, 1999) and *SHELXL2014* (Sheldrick, 2015).

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full crystallographic data

IUCrData (2016). **1**, x161963 [https://doi.org/10.1107/S2414314616019635]

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Crystal data

$C_{32}H_{48}O_8P_2S_2$
M_r = 686.76
Monoclinic, $P2_1/c$
a = 13.6507 (8) Å
b = 18.8828 (11) Å
c = 14.5033 (8) Å
β = 103.279 (5)°
V = 3638.5 (4) Å^3
Z = 4
$F(000)$ = 1464
D_x = 1.254 Mg m^{-3}
Melting point: 458 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 17095 reflections
θ = 3.3–28.7°
μ = 0.28 mm^{-1}
T = 248 K
Needle, yellow
0.40 × 0.15 × 0.08 mm

Data collection

Stoe IPDS 2T diffractometer
Graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
25084 measured reflections
8750 independent reflections
3346 reflections with $I > 2\sigma(I)$
R_{int} = 0.143
θ_{max} = 28.0°, θ_{min} = 3.3°
h = −18→18
k = −24→24
l = −18→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.091
$wR(F^2)$ = 0.304
S = 0.95
8750 reflections
433 parameters
22 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.149P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.7646 (4)	0.4573 (3)	0.1961 (4)	0.0391 (13)	
C2	0.7995 (5)	0.4363 (3)	0.2908 (4)	0.0416 (14)	
H2	0.8312	0.3921	0.3046	0.050*	
C3	0.7876 (4)	0.4802 (3)	0.3644 (4)	0.0420 (14)	
C4	0.7430 (4)	0.5466 (3)	0.3458 (4)	0.0390 (14)	
C5	0.7098 (5)	0.5673 (3)	0.2520 (5)	0.0469 (15)	
H5	0.6797	0.6120	0.2383	0.056*	
C6	0.7200 (5)	0.5238 (3)	0.1788 (5)	0.0451 (15)	
O7	0.8192 (4)	0.4617 (2)	0.4578 (3)	0.0522 (12)	
C8	0.8661 (5)	0.3944 (3)	0.4836 (5)	0.0507 (16)	
H8A	0.8313	0.3585	0.4391	0.061*	
H8B	0.8578	0.3816	0.5469	0.061*	
C9	0.9747 (5)	0.3929 (4)	0.4839 (6)	0.0610 (19)	
H9A	1.0099	0.4297	0.5266	0.073*	
H9B	0.9836	0.4031	0.4201	0.073*	
C10	1.0204 (6)	0.3206 (5)	0.5163 (7)	0.077 (3)	
H10A	1.0116	0.3106	0.5794	0.116*	
H10B	1.0916	0.3210	0.5170	0.116*	
H10C	0.9870	0.2844	0.4728	0.116*	
O11	0.6883 (4)	0.5425 (3)	0.0852 (3)	0.0596 (13)	
C12	0.6181 (7)	0.6006 (5)	0.0622 (5)	0.073 (2)	
H12A	0.6526	0.6459	0.0788	0.087*	0.75 (2)
H12B	0.5647	0.5962	0.0970	0.087*	0.75 (2)
H12C	0.6481	0.6432	0.0955	0.087*	0.25 (2)
H12D	0.5571	0.5894	0.0842	0.087*	0.25 (2)
C13	0.5740 (17)	0.5964 (10)	−0.0441 (9)	0.101 (7)	0.75 (2)
H13A	0.5493	0.5484	−0.0616	0.121*	0.75 (2)
H13B	0.6259	0.6076	−0.0787	0.121*	0.75 (2)
C14	0.4868 (10)	0.6498 (9)	−0.0698 (12)	0.120 (7)	0.75 (2)
H14A	0.4578	0.6479	−0.1374	0.179*	0.75 (2)
H14B	0.4357	0.6381	−0.0356	0.179*	0.75 (2)
H14C	0.5121	0.6972	−0.0527	0.179*	0.75 (2)
C13A	0.590 (4)	0.615 (2)	−0.0446 (15)	0.057 (9)	0.25 (2)
H13C	0.6494	0.6148	−0.0711	0.068*	0.25 (2)
H13D	0.5565	0.6615	−0.0575	0.068*	0.25 (2)
C14A	0.517 (4)	0.555 (2)	−0.087 (2)	0.12 (2)	0.25 (2)
H14D	0.4960	0.5617	−0.1554	0.173*	0.25 (2)
H14E	0.5509	0.5101	−0.0736	0.173*	0.25 (2)
H14F	0.4585	0.5566	−0.0601	0.173*	0.25 (2)
C15	0.7770 (5)	0.4074 (3)	0.1201 (5)	0.0421 (14)	
S16	0.78638 (15)	0.43527 (9)	0.00974 (12)	0.0519 (5)	
C17	0.8043 (5)	0.3503 (4)	−0.0255 (5)	0.0456 (15)	
C18	0.8015 (5)	0.3034 (4)	0.0459 (5)	0.0504 (16)	
H18	0.8087	0.2542	0.0399	0.060*	
C19	0.7871 (5)	0.3356 (3)	0.1282 (5)	0.0469 (15)	

H19	0.7845	0.3104	0.1836	0.056*	
P20	0.82830 (13)	0.33165 (9)	−0.13808 (12)	0.0447 (4)	
O21	0.9359 (4)	0.3627 (3)	−0.1288 (4)	0.0601 (13)	
C22	0.9893 (6)	0.3613 (5)	−0.2057 (6)	0.072 (2)	
H22	0.9536	0.3291	−0.2563	0.087*	
C23	1.0921 (7)	0.3323 (6)	−0.1643 (9)	0.106 (4)	
H23A	1.1247	0.3609	−0.1104	0.159*	
H23B	1.1318	0.3333	−0.2118	0.159*	
H23C	1.0863	0.2839	−0.1438	0.159*	
C24	0.9899 (8)	0.4339 (6)	−0.2448 (8)	0.105 (4)	
H24A	0.9214	0.4484	−0.2730	0.158*	
H24B	1.0292	0.4343	−0.2927	0.158*	
H24C	1.0195	0.4664	−0.1943	0.158*	
O25	0.7626 (3)	0.3838 (3)	−0.2104 (3)	0.0537 (12)	
C26	0.6563 (6)	0.3737 (5)	−0.2468 (6)	0.072 (2)	
H26	0.6280	0.3364	−0.2126	0.087*	0.5
H26A	0.6355	0.3462	−0.1964	0.087*	0.5
C27	0.6073 (11)	0.4452 (8)	−0.2428 (11)	0.154 (6)	
H27A	0.6252	0.4765	−0.2894	0.230*	
H27B	0.6306	0.4653	−0.1801	0.230*	
H27C	0.5348	0.4395	−0.2565	0.230*	
C28	0.640 (3)	0.358 (2)	−0.3540 (18)	0.121 (12)	0.5
H28A	0.5694	0.3511	−0.3814	0.182*	0.5
H28B	0.6646	0.3980	−0.3848	0.182*	0.5
H28C	0.6775	0.3159	−0.3630	0.182*	0.5
C28A	0.630 (2)	0.3329 (17)	−0.3222 (18)	0.086 (7)	0.5
H28D	0.5572	0.3297	−0.3407	0.130*	0.5
H28E	0.6552	0.3531	−0.3738	0.130*	0.5
H28F	0.6582	0.2860	−0.3076	0.130*	0.5
O29	0.8151 (4)	0.2567 (2)	−0.1632 (3)	0.0555 (12)	
C30	0.7278 (4)	0.5954 (3)	0.4202 (4)	0.0397 (13)	
S31	0.73883 (13)	0.57008 (9)	0.53735 (12)	0.0474 (4)	
C32	0.7074 (4)	0.6532 (3)	0.5694 (4)	0.0403 (13)	
C33	0.6920 (5)	0.6977 (4)	0.4946 (5)	0.0503 (16)	
H33	0.6757	0.7458	0.4985	0.060*	
C34	0.7026 (5)	0.6654 (4)	0.4104 (5)	0.0509 (16)	
H34	0.6933	0.6897	0.3525	0.061*	
P35	0.68713 (13)	0.67232 (9)	0.68365 (12)	0.0453 (4)	
O36	0.7622 (4)	0.6266 (3)	0.7574 (4)	0.0626 (13)	
C37	0.8578 (6)	0.6543 (5)	0.8129 (6)	0.073 (2)	
H37	0.8513	0.7055	0.8254	0.088*	
C38	0.8805 (9)	0.6139 (11)	0.9042 (9)	0.178 (8)	
H38A	0.8315	0.6259	0.9407	0.267*	
H38B	0.9474	0.6260	0.9402	0.267*	
H38C	0.8774	0.5635	0.8909	0.267*	
C39	0.9374 (8)	0.6428 (8)	0.7584 (10)	0.128 (5)	
H39A	0.9446	0.5925	0.7483	0.192*	
H39B	1.0009	0.6618	0.7939	0.192*	

H39C	0.9182	0.6666	0.6976	0.192*
O40	0.5887 (3)	0.6316 (3)	0.6897 (4)	0.0566 (12)
C41	0.4946 (6)	0.6448 (5)	0.6224 (6)	0.074 (2)
H41	0.5103	0.6647	0.5643	0.089*
C42	0.4431 (8)	0.5766 (7)	0.5975 (10)	0.120 (4)
H42A	0.4884	0.5437	0.5772	0.181*
H42B	0.3840	0.5837	0.5466	0.181*
H42C	0.4229	0.5575	0.6524	0.181*
C43	0.4360 (11)	0.6990 (9)	0.6620 (15)	0.207 (10)
H43A	0.4116	0.6785	0.7138	0.310*
H43B	0.3793	0.7146	0.6128	0.310*
H43C	0.4789	0.7392	0.6851	0.310*
O44	0.6885 (4)	0.7489 (2)	0.6998 (4)	0.0593 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.047 (3)	0.035 (3)	0.039 (3)	0.000 (3)	0.016 (3)	−0.002 (3)
C2	0.055 (4)	0.034 (3)	0.038 (3)	0.003 (3)	0.016 (3)	0.001 (3)
C3	0.049 (3)	0.041 (4)	0.040 (3)	−0.004 (3)	0.018 (3)	0.003 (3)
C4	0.046 (3)	0.033 (3)	0.041 (3)	0.002 (3)	0.018 (3)	0.000 (3)
C5	0.059 (4)	0.037 (4)	0.048 (4)	0.006 (3)	0.019 (3)	−0.001 (3)
C6	0.055 (4)	0.041 (4)	0.039 (3)	0.004 (3)	0.010 (3)	0.004 (3)
O7	0.077 (3)	0.039 (3)	0.043 (3)	0.016 (2)	0.019 (2)	0.001 (2)
C8	0.062 (4)	0.037 (4)	0.054 (4)	0.009 (3)	0.015 (3)	0.006 (3)
C9	0.055 (4)	0.059 (5)	0.071 (5)	0.001 (3)	0.019 (4)	0.000 (4)
C10	0.053 (4)	0.066 (6)	0.108 (8)	0.010 (4)	0.007 (5)	−0.007 (5)
O11	0.096 (4)	0.046 (3)	0.036 (2)	0.023 (3)	0.015 (2)	0.002 (2)
C12	0.105 (6)	0.058 (5)	0.049 (4)	0.033 (4)	0.004 (4)	−0.001 (4)
C13	0.155 (18)	0.061 (12)	0.072 (9)	0.018 (12)	−0.003 (9)	−0.001 (8)
C14	0.086 (10)	0.120 (14)	0.127 (14)	0.004 (8)	−0.028 (9)	0.051 (11)
C13A	0.07 (2)	0.051 (18)	0.044 (9)	0.019 (13)	0.006 (8)	−0.003 (8)
C14A	0.19 (4)	0.12 (3)	0.029 (12)	−0.07 (3)	0.008 (14)	0.010 (12)
C15	0.049 (3)	0.035 (3)	0.045 (4)	0.000 (3)	0.016 (3)	−0.003 (3)
S16	0.0842 (13)	0.0363 (9)	0.0418 (9)	0.0038 (8)	0.0284 (9)	0.0011 (7)
C17	0.047 (3)	0.044 (4)	0.049 (4)	0.000 (3)	0.018 (3)	−0.003 (3)
C18	0.063 (4)	0.040 (4)	0.052 (4)	0.004 (3)	0.021 (3)	−0.002 (3)
C19	0.066 (4)	0.040 (4)	0.040 (3)	0.004 (3)	0.023 (3)	0.001 (3)
P20	0.0555 (10)	0.0441 (10)	0.0386 (9)	0.0021 (8)	0.0190 (8)	−0.0022 (7)
O21	0.066 (3)	0.067 (3)	0.053 (3)	−0.006 (2)	0.026 (2)	−0.014 (3)
C22	0.078 (5)	0.083 (6)	0.068 (5)	−0.027 (5)	0.041 (4)	−0.020 (5)
C23	0.085 (7)	0.112 (9)	0.143 (10)	0.016 (6)	0.071 (7)	0.001 (7)
C24	0.120 (8)	0.106 (9)	0.101 (8)	−0.031 (7)	0.049 (7)	0.014 (7)
O25	0.065 (3)	0.058 (3)	0.040 (3)	0.008 (2)	0.015 (2)	0.006 (2)
C26	0.072 (5)	0.082 (6)	0.063 (5)	0.002 (4)	0.015 (4)	0.016 (4)
C27	0.149 (11)	0.142 (9)	0.151 (13)	0.062 (8)	−0.006 (9)	−0.022 (8)
C28	0.15 (2)	0.16 (3)	0.061 (16)	0.02 (2)	0.032 (16)	0.002 (17)
C28A	0.079 (13)	0.105 (13)	0.073 (11)	0.015 (11)	0.012 (10)	0.006 (10)

O29	0.081 (3)	0.045 (3)	0.045 (3)	0.000 (2)	0.023 (2)	−0.006 (2)
C30	0.044 (3)	0.041 (4)	0.035 (3)	0.002 (3)	0.013 (3)	−0.002 (3)
S31	0.0663 (10)	0.0374 (9)	0.0421 (9)	0.0044 (7)	0.0200 (8)	−0.0031 (7)
C32	0.048 (3)	0.038 (3)	0.036 (3)	0.001 (3)	0.011 (3)	−0.005 (3)
C33	0.065 (4)	0.036 (4)	0.054 (4)	0.006 (3)	0.024 (3)	−0.009 (3)
C34	0.065 (4)	0.042 (4)	0.051 (4)	0.009 (3)	0.022 (3)	0.002 (3)
P35	0.0516 (10)	0.0427 (10)	0.0433 (9)	0.0008 (7)	0.0145 (8)	−0.0068 (8)
O36	0.064 (3)	0.053 (3)	0.066 (3)	0.002 (2)	0.004 (3)	−0.002 (3)
C37	0.057 (4)	0.094 (7)	0.060 (5)	−0.008 (4)	−0.005 (4)	−0.002 (5)
C38	0.096 (9)	0.34 (2)	0.075 (8)	−0.011 (12)	−0.030 (7)	0.013 (12)
C39	0.069 (6)	0.163 (13)	0.159 (13)	0.008 (7)	0.039 (7)	0.007 (10)
O40	0.059 (3)	0.058 (3)	0.055 (3)	−0.004 (2)	0.019 (2)	−0.001 (2)
C41	0.060 (5)	0.096 (7)	0.067 (5)	−0.001 (4)	0.017 (4)	0.003 (5)
C42	0.079 (7)	0.144 (12)	0.127 (10)	−0.037 (7)	0.001 (6)	0.003 (8)
C43	0.111 (10)	0.207 (18)	0.29 (2)	0.079 (11)	0.014 (12)	−0.110 (16)
O44	0.085 (3)	0.039 (3)	0.060 (3)	−0.002 (2)	0.027 (3)	−0.010 (2)

Geometric parameters (Å, º)

C1—C6	1.393 (9)	C22—C23	1.498 (13)
C1—C2	1.404 (8)	C22—H22	0.9900
C1—C15	1.490 (8)	C23—H23A	0.9700
C2—C3	1.391 (8)	C23—H23B	0.9700
C2—H2	0.9400	C23—H23C	0.9700
C3—O7	1.369 (7)	C24—H24A	0.9700
C3—C4	1.394 (8)	C24—H24B	0.9700
C4—C5	1.386 (9)	C24—H24C	0.9700
C4—C30	1.470 (8)	O25—C26	1.437 (9)
C5—C6	1.374 (9)	C26—C28A	1.32 (3)
C5—H5	0.9400	C26—C27	1.513 (15)
C6—O11	1.373 (7)	C26—C28	1.55 (3)
O7—C8	1.433 (7)	C26—H26	0.9900
C8—C9	1.482 (9)	C26—H26A	0.9900
C8—H8A	0.9800	C27—H27A	0.9700
C8—H8B	0.9800	C27—H27B	0.9700
C9—C10	1.530 (11)	C27—H27C	0.9700
C9—H9A	0.9800	C28—H28A	0.9700
C9—H9B	0.9800	C28—H28B	0.9700
C10—H10A	0.9700	C28—H28C	0.9700
C10—H10B	0.9700	C28A—H28D	0.9700
C10—H10C	0.9700	C28A—H28E	0.9700
O11—C12	1.444 (8)	C28A—H28F	0.9700
C12—C13	1.523 (13)	C30—C34	1.363 (9)
C12—C13A	1.534 (19)	C30—S31	1.737 (6)
C12—H12A	0.9800	S31—C32	1.719 (6)
C12—H12B	0.9800	C32—C33	1.350 (9)
C12—H12C	0.9800	C32—P35	1.778 (6)
C12—H12D	0.9800	C33—C34	1.401 (9)

C13—C14	1.540 (17)	C33—H33	0.9400
C13—H13A	0.9800	C34—H34	0.9400
C13—H13B	0.9800	P35—O44	1.464 (5)
C14—H14A	0.9700	P35—O36	1.561 (5)
C14—H14B	0.9700	P35—O40	1.569 (5)
C14—H14C	0.9700	O36—C37	1.464 (9)
C13A—C14A	1.54 (2)	C37—C38	1.499 (16)
C13A—H13C	0.9800	C37—C39	1.499 (13)
C13A—H13D	0.9800	C37—H37	0.9900
C14A—H14D	0.9700	C38—H38A	0.9700
C14A—H14E	0.9700	C38—H38B	0.9700
C14A—H14F	0.9700	C38—H38C	0.9700
C15—C19	1.366 (9)	C39—H39A	0.9700
C15—S16	1.717 (6)	C39—H39B	0.9700
S16—C17	1.717 (7)	C39—H39C	0.9700
C17—C18	1.370 (9)	O40—C41	1.445 (10)
C17—P20	1.774 (6)	C41—C42	1.473 (14)
C18—C19	1.394 (9)	C41—C43	1.493 (14)
C18—H18	0.9400	C41—H41	0.9900
C19—H19	0.9400	C42—H42A	0.9700
P20—O29	1.461 (5)	C42—H42B	0.9700
P20—O21	1.558 (5)	C42—H42C	0.9700
P20—O25	1.562 (5)	C43—H43A	0.9700
O21—C22	1.465 (8)	C43—H43B	0.9700
C22—C24	1.484 (13)	C43—H43C	0.9700

C6—C1—C2	117.9 (6)	C22—C23—H23A	109.5
C6—C1—C15	123.8 (6)	C22—C23—H23B	109.5
C2—C1—C15	118.3 (5)	H23A—C23—H23B	109.5
C3—C2—C1	120.6 (6)	C22—C23—H23C	109.5
C3—C2—H2	119.7	H23A—C23—H23C	109.5
C1—C2—H2	119.7	H23B—C23—H23C	109.5
O7—C3—C2	122.8 (6)	C22—C24—H24A	109.5
O7—C3—C4	116.5 (5)	C22—C24—H24B	109.5
C2—C3—C4	120.7 (6)	H24A—C24—H24B	109.5
C5—C4—C3	118.2 (6)	C22—C24—H24C	109.5
C5—C4—C30	118.3 (5)	H24A—C24—H24C	109.5
C3—C4—C30	123.4 (6)	H24B—C24—H24C	109.5
C6—C5—C4	121.5 (6)	C26—O25—P20	122.6 (5)
C6—C5—H5	119.3	C28A—C26—O25	116.2 (14)
C4—C5—H5	119.3	C28A—C26—C27	120.9 (15)
O11—C6—C5	123.1 (6)	O25—C26—C27	106.6 (9)
O11—C6—C1	115.8 (5)	O25—C26—C28	106.9 (17)
C5—C6—C1	121.1 (6)	C27—C26—C28	104.2 (17)
C3—O7—C8	120.3 (5)	O25—C26—H26	112.9
O7—C8—C9	113.8 (6)	C27—C26—H26	112.9
O7—C8—H8A	108.8	C28—C26—H26	112.9
C9—C8—H8A	108.8	C28A—C26—H26A	103.6

O7—C8—H8B	108.8	O25—C26—H26A	103.6
C9—C8—H8B	108.8	C27—C26—H26A	103.6
H8A—C8—H8B	107.7	C26—C27—H27A	109.5
C8—C9—C10	110.8 (6)	C26—C27—H27B	109.5
C8—C9—H9A	109.5	H27A—C27—H27B	109.5
C10—C9—H9A	109.5	C26—C27—H27C	109.5
C8—C9—H9B	109.5	H27A—C27—H27C	109.5
C10—C9—H9B	109.5	H27B—C27—H27C	109.5
H9A—C9—H9B	108.1	C26—C28—H28A	109.5
C9—C10—H10A	109.5	C26—C28—H28B	109.5
C9—C10—H10B	109.5	H28A—C28—H28B	109.5
H10A—C10—H10B	109.5	C26—C28—H28C	109.5
C9—C10—H10C	109.5	H28A—C28—H28C	109.5
H10A—C10—H10C	109.5	H28B—C28—H28C	109.5
H10B—C10—H10C	109.5	C26—C28A—H28D	109.5
C6—O11—C12	117.7 (5)	C26—C28A—H28E	109.5
O11—C12—C13	106.3 (9)	H28D—C28A—H28E	109.5
O11—C12—C13A	112 (2)	C26—C28A—H28F	109.5
O11—C12—H12A	110.5	H28D—C28A—H28F	109.5
C13—C12—H12A	110.5	H28E—C28A—H28F	109.5
O11—C12—H12B	110.5	C34—C30—C4	127.2 (6)
C13—C12—H12B	110.5	C34—C30—S31	109.4 (5)
H12A—C12—H12B	108.7	C4—C30—S31	123.4 (5)
O11—C12—H12C	109.3	C32—S31—C30	92.4 (3)
C13A—C12—H12C	109.3	C33—C32—S31	110.6 (5)
O11—C12—H12D	109.3	C33—C32—P35	126.5 (5)
C13A—C12—H12D	109.3	S31—C32—P35	122.6 (4)
H12C—C12—H12D	107.9	C32—C33—C34	113.7 (6)
C12—C13—C14	108.3 (13)	C32—C33—H33	123.2
C12—C13—H13A	110.0	C34—C33—H33	123.2
C14—C13—H13A	110.0	C30—C34—C33	113.9 (6)
C12—C13—H13B	110.0	C30—C34—H34	123.1
C14—C13—H13B	110.0	C33—C34—H34	123.1
H13A—C13—H13B	108.4	O44—P35—O36	117.0 (3)
C13—C14—H14A	109.5	O44—P35—O40	117.0 (3)
C13—C14—H14B	109.5	O36—P35—O40	97.3 (3)
H14A—C14—H14B	109.5	O44—P35—C32	110.5 (3)
C13—C14—H14C	109.5	O36—P35—C32	107.9 (3)
H14A—C14—H14C	109.5	O40—P35—C32	105.7 (3)
H14B—C14—H14C	109.5	C37—O36—P35	122.7 (5)
C12—C13A—C14A	105 (3)	O36—C37—C38	106.3 (8)
C12—C13A—H13C	110.7	O36—C37—C39	108.7 (8)
C14A—C13A—H13C	110.7	C38—C37—C39	111.3 (10)
C12—C13A—H13D	110.7	O36—C37—H37	110.2
C14A—C13A—H13D	110.7	C38—C37—H37	110.2
H13C—C13A—H13D	108.8	C39—C37—H37	110.2
C13A—C14A—H14D	109.5	C37—C38—H38A	109.5
C13A—C14A—H14E	109.5	C37—C38—H38B	109.5

H14D—C14A—H14E	109.5	H38A—C38—H38B	109.5
C13A—C14A—H14F	109.5	C37—C38—H38C	109.5
H14D—C14A—H14F	109.5	H38A—C38—H38C	109.5
H14E—C14A—H14F	109.5	H38B—C38—H38C	109.5
C19—C15—C1	126.2 (6)	C37—C39—H39A	109.5
C19—C15—S16	111.0 (5)	C37—C39—H39B	109.5
C1—C15—S16	122.8 (5)	H39A—C39—H39B	109.5
C15—S16—C17	92.1 (3)	C37—C39—H39C	109.5
C18—C17—S16	110.5 (5)	H39A—C39—H39C	109.5
C18—C17—P20	127.6 (5)	H39B—C39—H39C	109.5
S16—C17—P20	121.9 (4)	C41—O40—P35	121.1 (5)
C17—C18—C19	113.5 (6)	O40—C41—C42	108.3 (8)
C17—C18—H18	123.3	O40—C41—C43	109.3 (9)
C19—C18—H18	123.3	C42—C41—C43	115.2 (10)
C15—C19—C18	113.0 (6)	O40—C41—H41	107.9
C15—C19—H19	123.5	C42—C41—H41	107.9
C18—C19—H19	123.5	C43—C41—H41	107.9
O29—P20—O21	116.4 (3)	C41—C42—H42A	109.5
O29—P20—O25	115.0 (3)	C41—C42—H42B	109.5
O21—P20—O25	101.9 (3)	H42A—C42—H42B	109.5
O29—P20—C17	112.6 (3)	C41—C42—H42C	109.5
O21—P20—C17	102.5 (3)	H42A—C42—H42C	109.5
O25—P20—C17	107.1 (3)	H42B—C42—H42C	109.5
C22—O21—P20	123.5 (5)	C41—C43—H43A	109.5
O21—C22—C24	108.8 (7)	C41—C43—H43B	109.5
O21—C22—C23	106.6 (8)	H43A—C43—H43B	109.5
C24—C22—C23	113.8 (8)	C41—C43—H43C	109.5
O21—C22—H22	109.2	H43A—C43—H43C	109.5
C24—C22—H22	109.2	H43B—C43—H43C	109.5
C23—C22—H22	109.2		

C6—C1—C2—C3	1.3 (9)	C18—C17—P20—O25	145.5 (6)
C15—C1—C2—C3	−178.4 (5)	S16—C17—P20—O25	−37.6 (5)
C1—C2—C3—O7	179.3 (5)	O29—P20—O21—C22	57.9 (7)
C1—C2—C3—C4	−1.6 (9)	O25—P20—O21—C22	−68.1 (6)
O7—C3—C4—C5	−179.9 (5)	C17—P20—O21—C22	−178.9 (6)
C2—C3—C4—C5	0.9 (9)	P20—O21—C22—C24	106.5 (8)
O7—C3—C4—C30	−1.1 (8)	P20—O21—C22—C23	−130.4 (7)
C2—C3—C4—C30	179.7 (6)	O29—P20—O25—C26	48.7 (6)
C3—C4—C5—C6	0.1 (9)	O21—P20—O25—C26	175.6 (6)
C30—C4—C5—C6	−178.8 (6)	C17—P20—O25—C26	−77.2 (6)
C4—C5—C6—O11	−179.8 (6)	P20—O25—C26—C28A	−86.8 (15)
C4—C5—C6—C1	−0.3 (10)	P20—O25—C26—C27	135.2 (8)
C2—C1—C6—O11	179.1 (6)	P20—O25—C26—C28	−113.9 (15)
C15—C1—C6—O11	−1.2 (9)	C5—C4—C30—C34	−14.3 (10)
C2—C1—C6—C5	−0.3 (9)	C3—C4—C30—C34	166.9 (6)
C15—C1—C6—C5	179.4 (6)	C5—C4—C30—S31	165.1 (5)
C2—C3—O7—C8	−0.7 (9)	C3—C4—C30—S31	−13.7 (8)

C4—C3—O7—C8	−179.9 (5)	C34—C30—S31—C32	1.0 (5)
C3—O7—C8—C9	82.2 (8)	C4—C30—S31—C32	−178.5 (5)
O7—C8—C9—C10	177.5 (6)	C30—S31—C32—C33	−1.5 (5)
C5—C6—O11—C12	−17.3 (10)	C30—S31—C32—P35	173.0 (4)
C1—C6—O11—C12	163.3 (7)	S31—C32—C33—C34	1.6 (8)
C6—O11—C12—C13	−164.4 (11)	P35—C32—C33—C34	−172.6 (5)
C6—O11—C12—C13A	−179.9 (18)	C4—C30—C34—C33	179.1 (6)
O11—C12—C13—C14	171.3 (13)	S31—C30—C34—C33	−0.3 (8)
O11—C12—C13A—C14A	75 (4)	C32—C33—C34—C30	−0.8 (9)
C6—C1—C15—C19	−156.2 (7)	C33—C32—P35—O44	−21.7 (7)
C2—C1—C15—C19	23.5 (9)	S31—C32—P35—O44	164.7 (4)
C6—C1—C15—S16	27.1 (8)	C33—C32—P35—O36	−150.8 (6)
C2—C1—C15—S16	−153.2 (5)	S31—C32—P35—O36	35.6 (5)
C19—C15—S16—C17	−0.2 (5)	C33—C32—P35—O40	105.9 (6)
C1—C15—S16—C17	177.0 (5)	S31—C32—P35—O40	−67.7 (4)
C15—S16—C17—C18	0.7 (5)	O44—P35—O36—C37	−27.5 (7)
C15—S16—C17—P20	−176.6 (4)	O40—P35—O36—C37	−153.0 (6)
S16—C17—C18—C19	−1.1 (8)	C32—P35—O36—C37	97.8 (6)
P20—C17—C18—C19	176.1 (5)	P35—O36—C37—C38	150.9 (9)
C1—C15—C19—C18	−177.5 (6)	P35—O36—C37—C39	−89.1 (9)
S16—C15—C19—C18	−0.4 (8)	O44—P35—O40—C41	65.1 (6)
C17—C18—C19—C15	1.0 (9)	O36—P35—O40—C41	−169.5 (6)
C18—C17—P20—O29	18.1 (7)	C32—P35—O40—C41	−58.5 (6)
S16—C17—P20—O29	−165.0 (4)	P35—O40—C41—C42	139.7 (7)
C18—C17—P20—O21	−107.7 (6)	P35—O40—C41—C43	−94.0 (11)
S16—C17—P20—O21	69.2 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O29^{i}	0.98	2.61	3.533 (9)	156
C12—H12*C*···O44^{ii}	0.98	2.52	3.480 (10)	166
C19—H19···O29^{i}	0.94	2.51	3.436 (8)	169
C34—H34···O44^{ii}	0.94	2.49	3.424 (8)	174

Symmetry codes: (i) x, $-y+1/2$, $z+1/2$; (ii) x, $-y+3/2$, $z-1/2$.