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A 1:2 co-crystal of 2,2′-thiodibenzoic acid and triphenylphosphane oxide: crystal structure, Hirshfeld surface analysis and computational study

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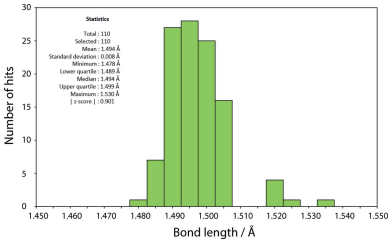
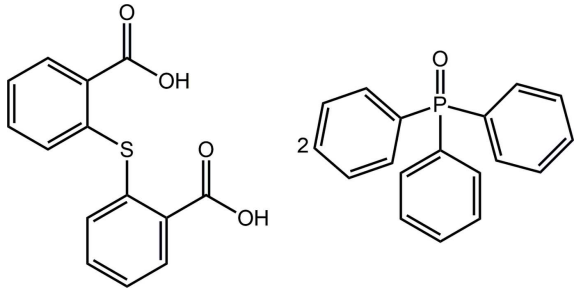
The asymmetric unit of the title co-crystal, 2,2′-thiodibenzoic acid–triphenylphosphane oxide (1/2), $C_{14}H_{10}O_4S\cdot 2C_{18}H_{15}OP$, comprises two molecules of 2,2′-thiodibenzoic acid [TDBA; systematic name: 2-[(2-carboxyphenyl)sulfanyl]benzoic acid] and four molecules of triphenylphosphane oxide [TPPO; systematic name: (diphenylphosphoryl)benzene]. The two TDBA molecules are twisted about their disulfide bonds and exhibit dihedral angles of 74.40 (5) and 72.58 (5)° between the planes through the two SC_6H_4 residues. The carboxylic acid groups are tilted out of the planes of the rings to which they are attached forming a range of CO_2/C_6 dihedral angles of 19.87 (6)–60.43 (8)°. Minor conformational changes are exhibited in the TPPO molecules with the range of dihedral angles between phenyl rings being −2.1 (1) to −62.8 (1)°. In the molecular packing, each TDBA acid molecule bridges two TPPO molecules *via* hydroxy-O—H···O(oxide) hydrogen bonds to form two three-molecule aggregates. These are connected into a three-dimensional architecture by TPPO-C—H···O(oxide, carbonyl) and TDBA-C—H···(oxide, carbonyl) interactions. The importance of H···H, O···H/H···O and C···H/H···C contacts to the calculated Hirshfeld surfaces has been demonstrated. In terms of individual molecules, O···H/H···O contacts are more important for the TDBA (*ca* 28%) than for the TPPO molecules (*ca* 13%), as expected from the chemical composition of these species. Computational chemistry indicates the four independent hydroxy-O—H···O(oxide) hydrogen bonds in the crystal impart about the same energy (*ca* 52 kJ mol^{-1}), with DTBA-phenyl-C—H···O(oxide) interactions being next most stabilizing (*ca* 40 kJ mol^{-1}).

1. Chemical context

2-Thiosalicylic acid, also known as 2-mercaptobenzoic acid, being an analogue to salicylic acid, has many applications. In medicine, is dianion is found in the salt $Na[EtHg(SC_6H_4CO_2)]$, which displays anti-fungal and anti-septic activities (Bigham & Copes, 2005). Other uses include as anti-corrosion agents (Chien *et al.*, 2012), as reactive agents or modifiers for nanoparticles and electrochemical sensing (Cang *et al.*, 2017; Sikarwar *et al.*, 2014), as catalysts for organic syntheses (Yang *et al.*, 2018; Selig & Miller, 2011) as well as being the precursor for some anti-viral and anti-microbial agents (Saha *et al.*, 2017). The compound readily coordinates a wide variety of metals, in both neutral and anionic form, due to the presence of both hard (oxygen) and soft (sulfur) donor atoms and exhibits different modes of coordination. Very recent reviews of the coordination chemistry of 2-thiosalicylic acid (Wehr-Candler & Henderson, 2016) and the isomeric 3- and 4-species (Tiekink & Henderson, 2017) are available. However, a restriction in the chemistry of this molecule is found as it can



undergo various pH-dependent transformations, *i.e.* it remains intact in acidic condition but may be oxidized to form 2,2′-dithiodibenzoic acid at neutral pH. For example and relevant to the present contribution, are studies of co-crystal formation between 2-thiosalicylic acid and bipyridyl-type molecules (Broker & Tiekink, 2007) whereby 2-thiosalicylic acid was oxidized to 2,2′-dithiodibenzoic acid during co-crystallization. During attempts to react 2-thiosalicylic acid with copper(I) chloride in the presence of two equivalents of triphenylphosphane, motivated by the desire to prepare analogues of phosphanecopper(I) dithiocarbamate derivatives which exhibit promising anti-bacterial activity (Jamaludin *et al.*, 2016), the title co-crystal was isolated, *i.e.* the 1:2 co-crystal of 2,2′-thiodibenzoic acid and triphenylphosphane oxide (I). Unexpectedly, both organic reagents were found to have oxidized in the presence of copper(I) chloride in acetonitrile solution under neutral conditions. While the actual mechanism remains unclear, a very recent study describes related synthetic outcomes (Gorobet *et al.*, 2018). Herein, the crystal and molecular structures, the analysis of the calculated Hirshfeld surface and calculation of the interaction energies through a computational approach for (I) are described.



2. Structural commentary

X-ray crystallography reveals the title co-crystal to comprise 2,2′-thiodibenzoic acid (TDBA) and triphenylphosphane oxide (TPPO) in the ratio 1:2, but with two independent TDBA molecules, Fig. 1, and four independent TPPO molecules, Fig. 2, in the asymmetric unit.

Each TDBA molecule comprises two benzoic acid residues connected in the 2-positions by a sulfur bridge. The confirmation of the presence of carboxylic acid groups is readily seen in the disparity in the C—O(hydroxy) and C═O(carbonyl) bond lengths with the minimum difference seen for the C100═O11 and C100—O12 bonds of 1.3126 (15) and 1.2075 (16) Å, respectively. As expected, the thiophenyl residues are almost planar with the maximum r.m.s. deviation of 0.053 Å being found for the S1,C80–C85 atoms. The thiophenyl rings are deviated from the perfect perpendicular bisector with dihedral angles of 74.40 (5) and 72.58 (5)° for the S1- and S2-molecules, respectively. Finally, the O6-, O8-, O10- and O12- carboxylic acid groups are tilted from the phenyl rings they are connected to by 60.43 (8), 24.24 (7), 19.87 (6) and 45.78 (7)°, respectively. That there are no major conformational differences between the molecules is evidenced from the overlay diagram of Fig. 3 (r.m.s. deviation = 0.118 Å).

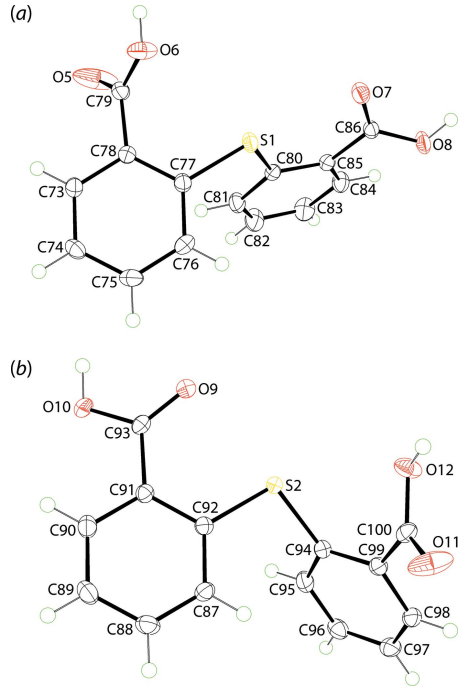


Figure 1
The molecular structures of the two independent molecules of 2,2′-thiodibenzoic acid in the asymmetric unit of (I), showing the atom-labelling scheme and displacement ellipsoids at the 70% probability level.

The molecular structures of the TPPO coformers are more rigid. This is seen in the O—P—C—C torsion angles, which range from 17.7 (1) to 61.6 (1), 19.8 (1) to 61.5 (1), −2.1 (1) to −62.8 (1) and −19.2 (1) to −44.5 (1)° for the P1–P4-molecules, respectively. In the same way, the P═O bond lengths span an experimentally equivalent range, *i.e.* 1.4975 (8) [P4═O4] to 1.5018 (8) Å [P1═O1].

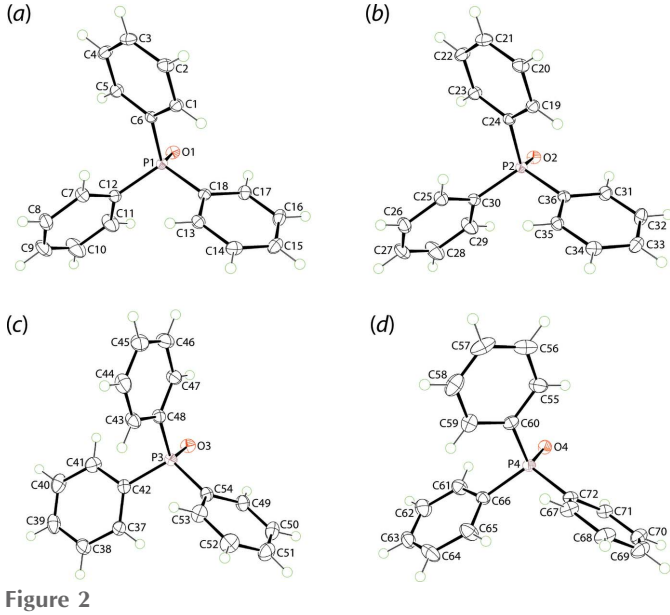


Figure 2
The molecular structures of the four independent molecules of triphenylphosphane oxide in the asymmetric unit of (I), showing the atom-labelling scheme and displacement ellipsoids at the 70% probability level.

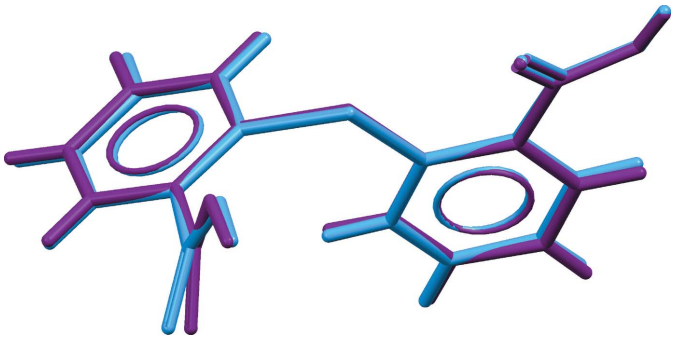


Figure 3
An overlay diagram of the two independent molecules of 2,2′-thiodibenzoic, with S1-molecule (purple) and S2-molecule (light-blue) superimposed so that a pair thiophenyl moieties are coincident.

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6*O*···O4	0.95 (2)	1.66 (2)	2.6070 (12)	171 (2)
O8—H8*O*···O1^{i}	0.90 (2)	1.70 (2)	2.5763 (12)	163 (2)
O10—H10*O*···O2^{ii}	0.91 (2)	1.72 (2)	2.6077 (12)	163 (2)
O12—H12*O*···O3^{iii}	0.90 (2)	1.71 (2)	2.5978 (12)	170.9 (19)
C16—H16···O4	0.93	2.53	3.3333 (15)	144
C44—H44···O4^{iv}	0.93	2.43	3.2404 (17)	145
C52—H52···O11^{v}	0.93	2.49	3.3231 (16)	149
C62—H62···O11^{i}	0.93	2.51	3.367 (2)	153
C64—H64···O5^{vi}	0.93	2.46	3.263 (2)	144
C68—H68···O3^{vi}	0.93	2.55	3.2747 (17)	135
C71—H71···O5	0.93	2.59	3.2765 (18)	131
C75—H75···O2^{i}	0.93	2.41	3.1184 (16)	133
C96—H96···O1	0.93	2.49	3.1832 (15)	132

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

3. Supramolecular features

Geometric parameters characterizing the identified (*PLATON*; Spek, 2009) interatomic contacts in the crystal of (I) are given in Table 1. The most prominent feature of the molecular packing is the formation of hydroxy-O—H···O(oxide) hydrogen bonds. These occur so that each molecule of 2,2′-thiodibenzoic acid (TDBA) links two triphenylphosphane oxide (TPPO) molecules to form a pair of three-molecule aggregates with a 13-membered, linear {O···HOC_3SC_3OH···O} heterosynthon as illustrated in Fig. 4. These aggregates are connected into a three-dimensional architecture by a large number of C—H···O interactions. Two of these contacts, *i.e.* TPPO-C47—H···O11(carbonyl) and TPPO-C71—H···O5(carbonyl), operate in concert with hydroxy-O12—H···O3(oxide) and hydroxy-O6—H···O4(oxide) hydrogen bonds, respectively, to close a nine-membered {HC_2PO···HOCO···} synthon. The C—H···O contacts are of the type TPPO-C—H···O(oxide, carbonyl) and TDBA-C—H···O(oxide, carbonyl), Table 1. In addition to participating in hydroxy-O—H···O(oxide) hydrogen bonds, each of the O1–O3 atoms of TPPO form an additional C—H···O(oxide) contact whereas the O4 atom participates in two such interactions. One carbonyl group of each TDBA molecule, *i.e.* the O5 and O11 atoms, participates in two C—H···O(carbonyl) interactions, leaving no formal role for the carbonyl-O7 and O9 atoms in the molecular packing. A view of the unit-cell contents is shown in Fig. 5.

In terms of distinguishing between molecules based on intermolecular contacts, the carbonyl-O5 atom of DTBA accepts C—H···O interactions from phenyl rings derived from TPPO and DTBA, whereas the carbonyl-O11 atom accepts contacts from TPPO only. The DPPO-O4 atom is distinct from the O1–O3 atoms based on the number of interactions it forms. In common with the O4 atom, O3 accepts a C—H···O interaction from TPPO, whereas each of the O1 and O2 participates in DTBA-C—H···O contacts.

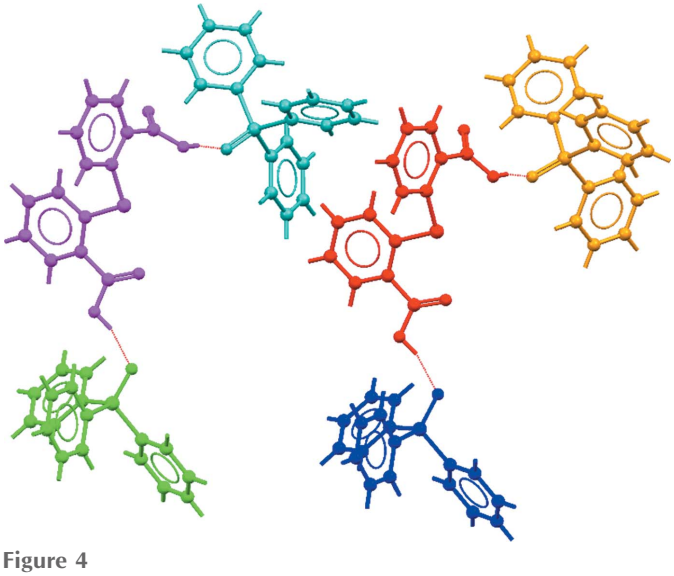


Figure 4
The two three-molecule aggregates in the crystal of (I). The hydroxy-O—H···O(oxide) hydrogen bonds are shown as red dashed lines. Colour code: S1-containing molecule, purple; S2, red; P1, green; P2, blue; P3, yellow; P4, light-blue.

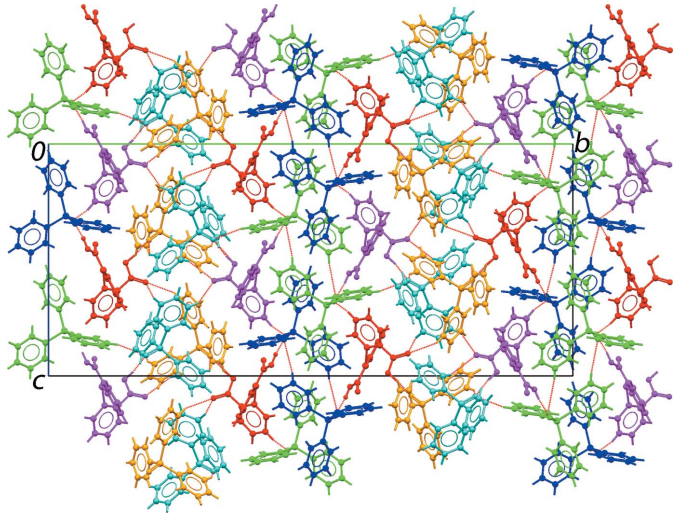


Figure 5
A view of the unit-cell contents shown in projection down the *a* axis. The molecules are colour-coded as for Fig. 4.

Table 2
Summary of short C···H interatomic contacts (Å) in (I).

Contact	Separation	Symmetry operation
C13···H10	2.80	$1-x, 1-y, 1-z$
C14···H10	2.84	$1-x, 1-y, 1-z$
C35···H28	2.77	$-x, 1-y, -z$
C34···H28	2.94	$-x, 1-y, -z$

4. Hirshfeld surface analysis

The independent 2,2′-thiodibenzoic acid (TDBA) and triphenylphosphane oxide (TPPO) molecules of (I) were subjected to Hirshfeld surface analysis following a literature precedent on a multi-component crystal (Jotani *et al.*, 2018) to further understand the nature of the intermolecular interactions in the crystal. As shown in Fig. 6(*a*)–(*f*), the pair of TDBA-S1 and -S2 molecules, shown with the respective pairs of hydrogen bonded TPPO molecules, as well as the TPPO-P1–P4 molecules exhibit some similarities especially on the prominent close contacts as represented by the intense red regions on the corresponding d_{norm} surface mappings, which are mainly dominated by hydroxy-O—H···O(oxide) interactions.

Upon close inspection on the surface mapping, minor differences are observed between the pair of TDBA molecules. Specifically, a diminutive red spot is observed near one of the terminal carboxylic groups of the S1-molecule arising from a TPPO-phenyl-C—H···O(carbonyl) interaction but, no such contact is apparent for the S2-molecule. As for the two pairs of TPPO molecules, the significant difference between the TPPO-P1 and -P4 molecules, linked to S1-DTBA, and the TPPO-P2 and P3 molecules, linked to the S2-TDBA, is the

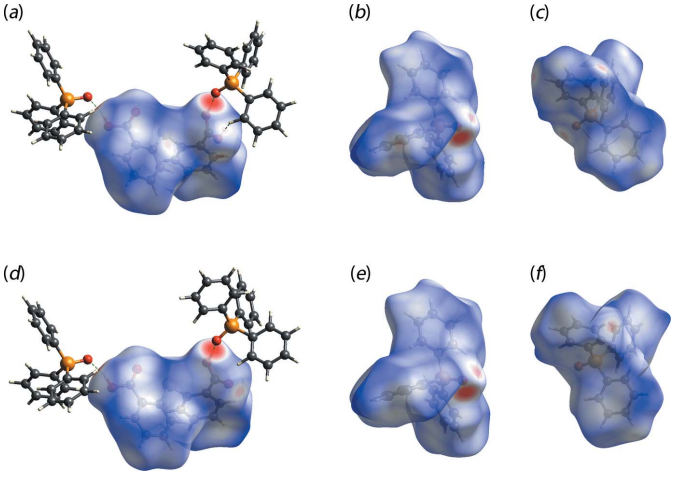


Figure 6
Views of the Hirshfeld surfaces mapped over d_{norm} for components of (I) for the: (*a*) S1-DTBA molecule hydrogen bonded (red dashed lines) to the P1- (left) and P4-TPPO molecules, (*b*) P1-TPPO, (*c*) P4-TPPO, (*d*) S2-DTBA molecule hydrogen bonded to the P2- (left) and P3-TPPO molecules, (*e*) P2-TPPO and (*f*) P3-TPPO. The surfaces in (*a*)–(*c*) are mapped over the range −0.766 to 1.446 a.u., and those in (*d*)–(*f*) over the range −0.766 to 1.563 a.u.

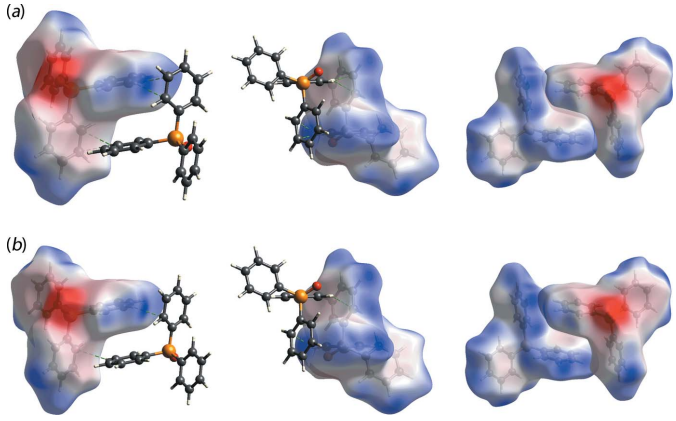


Figure 7
Different views of the Hirshfeld surfaces mapped over electrostatic potential for the centrosymmetrically related molecules of TPPO interacting *via* semi-localized phenyl-C—H···π(phenyl) interactions: (*a*) P1-TPPO, in the range of −0.100 to 0.041 a.u. and (*b*) P2-TPPO molecules (−0.100 to 0.041 a.u.).

presence of additional red spots on the surface mapping of the phenyl rings for P1- and P2-molecules in contrast to their P3- and P4-containing counterparts. This difference may be attributed to the complementary phenyl-C—H···π(phenyl) interactions between centrosymmetrically-related molecules, as illustrated in Fig. 7 and tabulated in Table 2. Here, the interacting H10 and H28 atoms are directed towards two carbon atoms of a symmetry-related ring so that the interactions are best described as being semi-localized as opposed to delocalized, which corresponds to the situation where the interacting hydrogen atom is equally separated from all six carbon atoms of the ring (Schollmeyer *et al.*, 2008).

Quantitative evaluation of the Hirshfeld surfaces by the combination of the d_i and d_e (*i* is internal and *e* is external to the surface) contact distances in intervals of 0.01 Å gives the overall two-dimensional fingerprint plots for the entire asymmetric unit of (I), Fig. 8(*a*), and each of the individual TDBA, Fig. 9(*a*), and TPPO, Fig. 10(*a*), molecules. Further, these can be delineated into specific contacts (McKinnon *et al.*, 2007) and Figs. 9–10(*b*)–(*d*) give fingerprint plots delineated into H···H, O···H/H···O and C···H/H···C contacts. The relative contributions of these contacts to the surfaces is given in Table 3.

The overall fingerprint plot for (I), Fig. 8*a*, is quite different for the individual components, Figs. 9–10*a*, as the former is a sum of all the individual surface contacts, which differ for the individual molecules. As expected, the same is true for the corresponding decomposed fingerprint plots. The major contribution to the overall surface of (I), *i.e.* 49.4%, comes from H···H contacts. The O···H/H···O contacts ($d_e + d_i \sim$ 2.34 Å) make a significant contribution at 13.7%, while the C···H/H···C interactions ($d_e + d_i \sim$ 2.66 Å), at 30.1%, play a more prominent role.

The formation of the 13-membered {O···HOC$_3$SC$_3$OH···O} heterosynthon, Fig. 4, is clearly reflected in the corresponding full fingerprint plots of the individual molecules Figs. 9–10(*a*), which exhibit an almost

Table 3
Percentage contributions of interatomic contacts to the Hirshfeld surface for (I) and for the the individual TDBA and DPPO molecules.

Contact	Percentage contribution						
	overall	S1-TDBA	S2-TDBA	P1-DPPO	P2-DPPO	P3-DPPO	P4-DPPO
H···H	49.4	42.3	40.7	49.8	49.6	49.7	51.4
O···H/H···O	13.7	28.1	28.1	14.1	13.6	11.7	12.7
C···H/H···C	30.1	21.9	23.4	30.2	30.9	33.4	31.3

identical claw-like fingerprint profile but arranged in the exact reverse order, *i.e.* Fig. 9(*a*) *cf.* Fig. 10(*a*). Among all the close interactions, H···H contacts, Figs. 8–9*b*, represent the dominant interactions to the individual surfaces, *i.e.* 41–42% for the TDBA molecules and 49–51% for the DPPO molecules, and exhibit $d_e + d_i$ contact distances ranging from 2.24 to 2.38 Å which is very close to the sum of van der Waals radii of 2.4 Å.

The O···H hydrogen bonds constitute the strongest among all interactions present in the co-crystal and lead to formation of asymmetric, forceps-like profiles in the corresponding decomposed fingerprint plots, Figs. 9–10(*c*). These feature two tips – one at relatively short $d_e + d_i$ ~1.6 Å that can be attributed to the hydroxy-H···O(oxide) hydrogen bonds for the S1- and S2-TDBA molecules, Fig. 10(*c*), or oxide-O···H(hydroxy) hydrogen bonds for P1–P4-TPPO. The other tip has a relatively long $d_e + d_i$ value of ~2.4 Å and arises as a result of hydroxy-O···H(phenyl) contacts for S1- and S2-TDBA or phenyl-H···O(hydroxy) for P1–P4-TPPO. The O···H/H···O contacts constitute the second most dominant interactions for the TDBA molecules and third most for the TPPO molecules, Table 3.

Similar to the H···H contacts, the C···H/H···C interactions contribute weakly to the molecular packing of the co-crystal as evidenced from the $d_e + d_i$ distance range of 2.7–2.8 Å, *i.e.* close to the sum of van der Waals radii of 2.9 Å, despite the contacts constituting the third most dominant interaction in the TDBA molecules (*ca* 22%) and being the second most dominant for the TPPO molecules (*ca* 32%). An exception to the trend is found for the P1- and P2-TPPO molecules, which display relatively short contact distances at *ca* 2.6 Å owing to the formation of C—H···π interactions as discussed above.

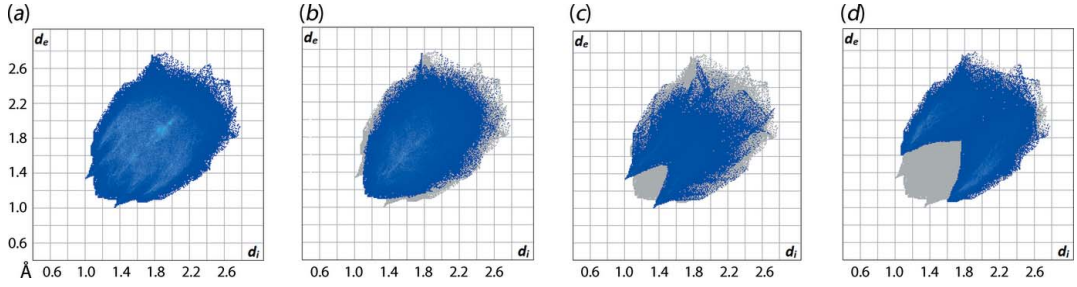


Figure 8
(*a*) The full two-dimensional fingerprint plot for (I) and (*b*)–(*d*) those delineated into H···H, O···H/H···O and C···H/H···C contacts, respectively.

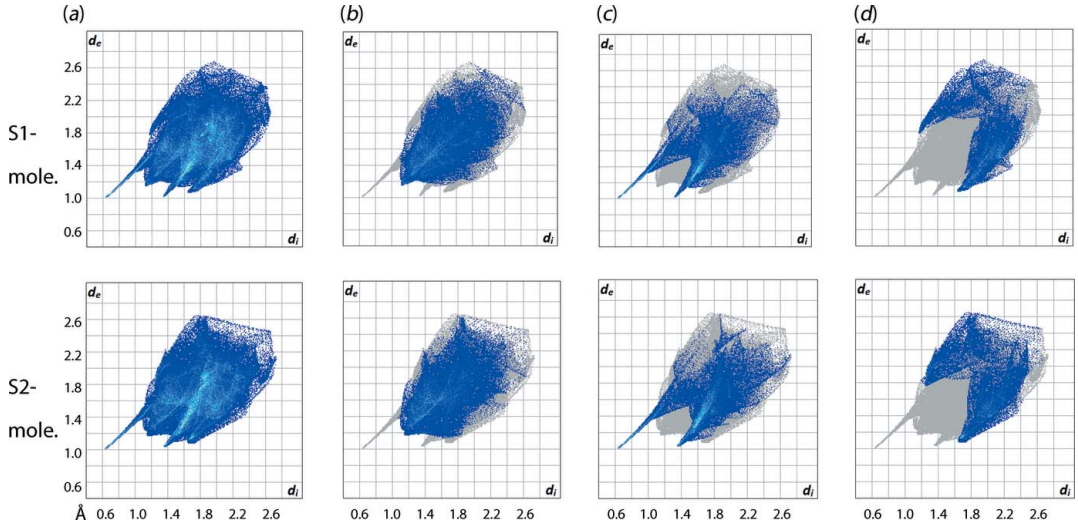


Figure 9
(*a*) The full two-dimensional fingerprint plot for the two independent TDBA molecules in (I) and (*b*)–(*d*) those delineated into H···H, O···H/H···O and C···H/H···C contacts, respectively.

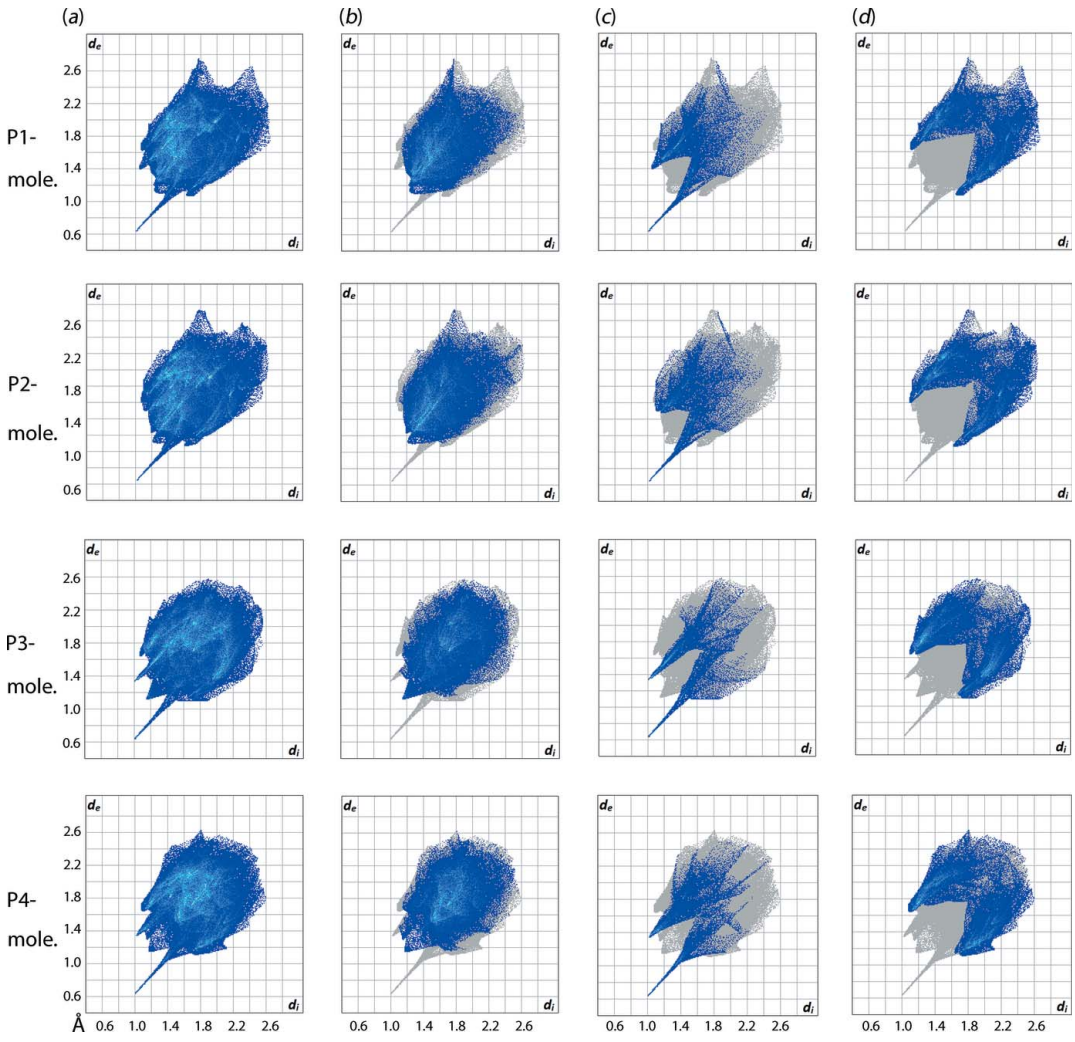


Figure 10
(*a*) The full two-dimensional fingerprint plot for the four independent TPPO molecules in (I) and (*b*)–(*d*) those delineated into H···H, O···H/H···O and C···H/H···C contacts, respectively.

In summary the Hirshfeld surface analysis on (I), with six individual constituents, was able to distinguish between these in terms of different intermolecular interactions, akin to the recently reported analysis of a structure with four independent cation/anion pairs (Jotani *et al.*, 2018).

5. Computational study

The co-crystal was subjected to intermolecular interaction energy calculations using CE-B3LYP/6-31G(*d*,*p*) available in *Crystal Explorer* (version 17; Turner *et al.*, 2017), with the crystal geometry being used as the input but, with hydrogen-atom positions normalized to the standard neutron diffraction values. By default, a cluster of molecules (defined as density matrices) would need to be generated by applying crystallographic symmetry operations with respect to a selected central molecule (density matrix) within the radius of 3.8 Å for interaction energy calculation (Turner *et al.*, 2014). However, as the co-crystal contains multiple independent molecules in the asymmetric unit, a cluster of molecules was first generated surrounding the S1-molecule of TDBA for the calculation and then the procedure was repeated for the cluster of molecules surrounding the S2-molecule. The total intermolecular energy is the sum of energies of four main components comprising electrostatic, polarization, dispersion and exchange-repulsion with a scale factors of 1.057, 0.740, 0.871 and 0.618, respectively (Mackenzie *et al.*, 2017).

Selected results obtained from the interaction energy calculations involving the DTBA molecules as reference molecules are tabulated in Table 4 and the environment about the S1-molecule of TDBA is shown in Fig. 11. As expected, O—H···O hydrogen bonding interactions give the greatest energies among the close contacts present in the crystal. The total intermolecular energy (E_{tot}) of the hydroxy-O—H···O(oxide) hydrogen bonds is consistent across the series and lies in the range −50.7 to −53.3 kJ mol^{-1}. The other close contacts which exerts a relatively strong influence in the energy frameworks of the co-crystal are DTBA-phenyl-C—H···O(oxide) interactions, with the E_{tot} amounting of *ca* −40 kJ mol^{-1}, Table 4.

Table 4
Interaction energies (kJ mol^{-1}) for selected close contacts.

contact	$E_{electrostatic}$	$E_{polarization}$	$E_{dispersion}$	$E_{exchange-repulsion}$	E_{total}	Symmetry operation
O6—H6*O*···O4	−76.5	−19.4	−17.8	95.2	−52.0	*x*, *y*, *z*
O8—H8*O*···O1	−72.8	−19.2	−14.9	82.3	−53.3	1 + *x*, *y*, *z*
O10—H10*O*···O2	−70.5	−18.3	−16.4	83.8	−50.7	1 + *x*, *y*, 1 + *z*
O12—H12*O*···O3	−72.3	−19.2	−13.9	81.2	−52.5	*x*, *y*, 1 + *z*
C75—H75···O2	−16.8	−6.6	−41.6	29.8	−40.4	1 + *x*, *y*, *z*
C96—H96···O1	−15.2	−6.1	−42.0	27.9	−40.0	*x*, *y*, *z*

6. Database survey

The only other structure of 2,2′-thiodibenzoic acid in the literature is that of the pure compound (Dai *et al.*, 2005). While this presents essentially the same features as for the two independent molecules in (I), the dihedral angle between the thiophenyl rings is up to 4° smaller at 68.0 (2)°, and the tilts of the carboxylic acid groups are less pronounced at 6.9 (5) and 29.8 (5)°.

A survey of the Cambridge Structural Database (Groom *et al.*, 2016), revealed 110 molecules of (non-coordinated) triphenylphosphane oxide. A plot of the retrieved P═O bond lengths is shown in Fig. 12. The mean value found for the P═O bond length is 1.494 Å with a standard deviation of 0.008 Å, with the minimum and maximum bond lengths being 1.478 (3) and 1.530 (7) Å, found in the multi-component structures of NUCHIC (Okawa *et al.*, 1997) and DUYXUQ (Arens *et al.*, 1986), respectively. In the latter structure, charge-assisted hydrogen bonds are formed between Ph_3P═O and Ph_3P═$O^{(+)}$H. The observed P═O bond lengths in (I), *i.e.* in the range 1.4975 (8) to 1.5018 (8) Å are at the lower end of the range of such bonds.

7. Synthesis and crystallization

All chemical precursors were of reagent grade and used as received without purification. Thiosalicylic acid (Merck; 0.154 g, 0.001 mol) and triphenylphosphane (Merck; 0.262 g, 0.002 mol) were dissolved in acetonitrile (40 ml) and the mixture subsequently added into an acetonitrile solution (25 ml) of copper(I) iodide (Merck; 0.19 g, 0.001 mol). The reaction mixture was stirred for 1 h at room temperature before the white product was filtered, washed with cold ethanol and dried *in vacuo*. The filtrate was left at room temperature, yielding colourless prisms after 1 week; Yield 74%. M.p. 457.7–459.2 K. IR (cm^{-1}): 3062 *ν*(C—H), 1693 *ν*(COO), 1236 *ν*(P═O), 1116 *ν*(P—Ar), 719 *δ*(P—C), 617 *ν*(C—S).

8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. The carbon-bound H atoms were placed in calculated positions (C—H = 0.93 Å) and were included in the refinement in the riding-model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). The oxygen-bound H atoms were located from difference Fourier maps and refined without constraint. Owing to poor agreement, three reflections, *i.e.* (1̄ 5 9), (3̄ 15 3) and (5̄ 7 9), were omitted from the final cycles of refinement.

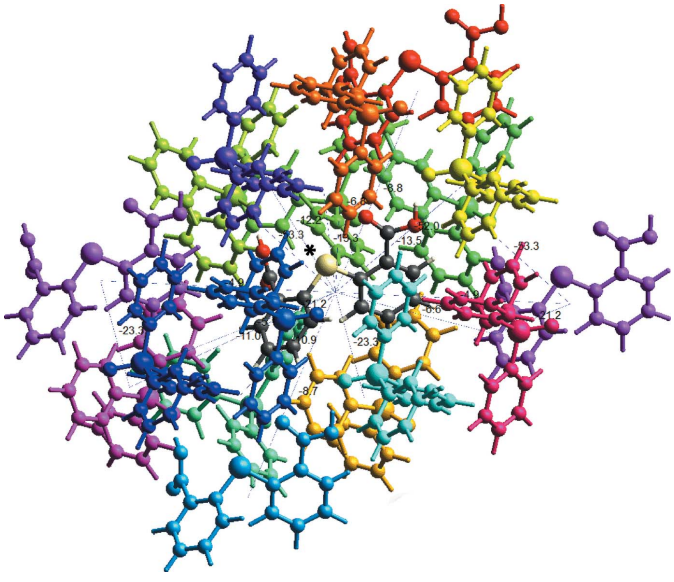


Figure 11
The interaction energy framework about the S1-molecule of DTBA (indicated by an asterisk) viewed along the *b*-axis direction.

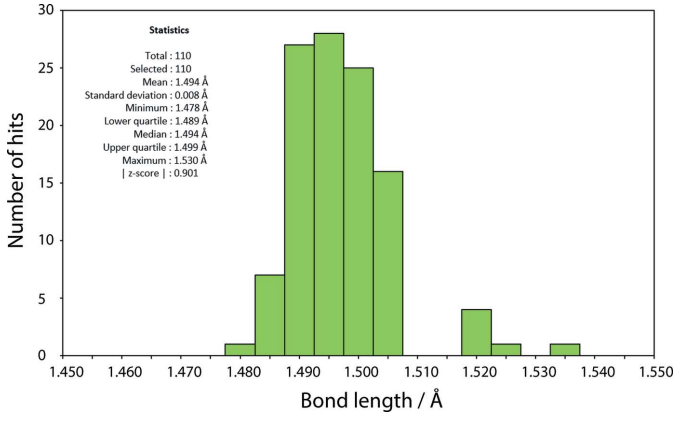


Figure 12
Statistical data on the P═O bond lengths as calculated by *Mogul* (Bruno *et al.*, 2004)

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Table 5
Experimental details.

Crystal data	
Chemical formula	$4C_{18}H_{15}OP{\cdot}2C_{14}H_{10}O_4S$
M_r	1661.64
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	10.7085 (1), 41.9751 (2), 18.9268 (1)
β (°)	101.490 (1)
V (Å^3)	8336.92 (10)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.83
Crystal size (mm)	0.17 × 0.16 × 0.09
Data collection	
Diffractometer	XtaLAB Synergy, Dualflex, AtlasS2
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2018)
T_{min}, T_{max}	0.653, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	94929, 17036, 15740
R_{int}	0.025
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.630
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.033, 0.092, 1.04
No. of reflections	17036
No. of parameters	1079
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.42, −0.55

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *OLEX2* (Dolomanov *et al*., 2009), *Mercury* (Macrae *et al.*, 2006) and *publCIF* (Westrip, 2010).

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supporting information

Acta Cryst. (2018). E**74**, 1764-1771 [https://doi.org/10.1107/S205698901801544X]

A 1:2 co-crystal of 2,2′-thiodibenzoic acid and triphenylphosphane oxide: crystal structure, Hirshfeld surface analysis and computational study

Sang Loon Tan and Edward R. T. Tiekink

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2018); cell refinement: *CrysAlis PRO* (Rigaku OD, 2018); data reduction: *CrysAlis PRO* (Rigaku OD, 2018); program(s) used to solve structure: SHELXT (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015*b*); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012), *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

2-[(2-Carboxyphenyl)sulfanyl]benzoic acid–(diphenylphosphoryl)benzene (1/2)

Crystal data

$4C_{18}H_{15}OP{\cdot}2C_{14}H_{10}O_4S$
M_r = 1661.64
Monoclinic, $P2_1/c$
a = 10.7085 (1) Å
b = 41.9751 (2) Å
c = 18.9268 (1) Å
β = 101.490 (1)°
V = 8336.92 (10) Å^3
Z = 4
$F(000)$ = 3472
D_x = 1.324 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 52739 reflections
θ = 4.2–75.9°
μ = 1.83 mm^{-1}
T = 100 K
Prism, colourless
0.17 × 0.16 × 0.09 mm

Data collection

XtaLAB Synergy, Dualflex, AtlasS2 diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
Detector resolution: 5.2558 pixels mm^{-1}
ω scans
Absorption correction: gaussian (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.653, T_{max} = 1.000
94929 measured reflections
17036 independent reflections
15740 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 76.2°, θ_{min} = 3.2°
h = −13→13
k = −49→52
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.092
S = 1.04
17036 reflections
1079 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 3.5049P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 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}
S1	0.86876 (3)	0.60239 (2)	0.45382 (2)	0.01933 (7)
O5	0.66647 (18)	0.68394 (3)	0.41031 (6)	0.0653 (5)
O6	0.63653 (11)	0.64495 (2)	0.48314 (5)	0.0298 (2)
H6O	0.644 (2)	0.6616 (5)	0.5178 (12)	0.064 (7)*
O7	1.05664 (8)	0.58755 (2)	0.57331 (5)	0.02348 (19)
O8	1.25159 (9)	0.57343 (2)	0.55791 (5)	0.0250 (2)
H8O	1.261 (2)	0.5646 (5)	0.6020 (12)	0.059 (6)*
C73	0.57062 (11)	0.63409 (3)	0.30175 (6)	0.0189 (2)
H73	0.510504	0.650346	0.296861	0.023*
C74	0.56910 (12)	0.61279 (3)	0.24561 (7)	0.0208 (2)
H74	0.507892	0.614647	0.203416	0.025*
C75	0.65935 (13)	0.58870 (3)	0.25272 (7)	0.0231 (3)
H75	0.659019	0.574453	0.215058	0.028*
C76	0.75002 (12)	0.58580 (3)	0.31576 (7)	0.0227 (3)
H76	0.809779	0.569467	0.320215	0.027*
C77	0.75263 (11)	0.60713 (3)	0.37271 (6)	0.0180 (2)
C78	0.66130 (11)	0.63139 (3)	0.36547 (6)	0.0161 (2)
C79	0.65763 (12)	0.65610 (3)	0.42226 (6)	0.0198 (2)
C80	1.01153 (11)	0.61285 (3)	0.42513 (6)	0.0167 (2)
C81	1.01083 (13)	0.62905 (3)	0.36024 (7)	0.0234 (3)
H81	0.933478	0.633723	0.329907	0.028*
C82	1.12341 (14)	0.63818 (4)	0.34059 (8)	0.0297 (3)
H82	1.120595	0.648892	0.297281	0.036*
C83	1.24037 (13)	0.63157 (4)	0.38468 (8)	0.0294 (3)
H83	1.315681	0.638162	0.371782	0.035*
C84	1.24298 (12)	0.61502 (3)	0.44811 (7)	0.0232 (3)
H84	1.321038	0.610079	0.477425	0.028*
C85	1.13031 (11)	0.60555 (3)	0.46905 (6)	0.0168 (2)
C86	1.14015 (11)	0.58810 (3)	0.53863 (6)	0.0170 (2)
S2	0.36308 (3)	0.60471 (2)	0.95618 (2)	0.01877 (7)
O9	0.56354 (8)	0.58971 (2)	1.06759 (4)	0.02008 (18)
O10	0.75481 (8)	0.57505 (2)	1.04690 (5)	0.02276 (19)
H10O	0.766 (2)	0.5668 (5)	1.0924 (12)	0.060 (6)*
O11	0.12548 (13)	0.68518 (2)	0.90397 (5)	0.0423 (3)
O12	0.15116 (10)	0.64745 (2)	0.98809 (5)	0.0254 (2)
H12O	0.1545 (18)	0.6638 (5)	1.0188 (11)	0.045 (5)*
C87	0.48876 (12)	0.62911 (3)	0.85303 (6)	0.0184 (2)

H87	0.408551	0.634762	0.827278	0.022*
C88	0.59558 (13)	0.63654 (3)	0.82546 (7)	0.0231 (3)
H88	0.586377	0.647289	0.781755	0.028*
C89	0.71638 (13)	0.62809 (3)	0.86246 (7)	0.0253 (3)
H89	0.787995	0.632986	0.843699	0.030*
C90	0.72868 (12)	0.61226 (3)	0.92771 (7)	0.0213 (2)
H90	0.809347	0.606453	0.952545	0.026*
C91	0.62200 (11)	0.60484 (3)	0.95702 (6)	0.0154 (2)
C92	0.49939 (11)	0.61319 (3)	0.91922 (6)	0.0151 (2)
C93	0.64177 (11)	0.58925 (3)	1.02911 (6)	0.0160 (2)
C94	0.24038 (11)	0.60750 (3)	0.87723 (6)	0.0179 (2)
C95	0.23738 (12)	0.58484 (3)	0.82279 (7)	0.0225 (3)
H95	0.297462	0.568554	0.828932	0.027*
C96	0.14576 (12)	0.58636 (3)	0.75969 (7)	0.0234 (3)
H96	0.145014	0.571214	0.723745	0.028*
C97	0.05526 (12)	0.61046 (3)	0.75020 (7)	0.0223 (3)
H97	−0.006374	0.611467	0.708014	0.027*
C98	0.05714 (11)	0.63302 (3)	0.80384 (6)	0.0194 (2)
H98	−0.003174	0.649254	0.797145	0.023*
C99	0.14835 (11)	0.63174 (3)	0.86788 (6)	0.0164 (2)
C100	0.14237 (11)	0.65763 (3)	0.92171 (6)	0.0181 (2)
P1	0.45306 (3)	0.53046 (2)	0.69428 (2)	0.01230 (6)
O1	0.32078 (8)	0.54411 (2)	0.67897 (4)	0.01763 (17)
C1	0.61145 (11)	0.53439 (3)	0.83070 (6)	0.0167 (2)
H1	0.656091	0.550177	0.811734	0.020*
C2	0.65061 (12)	0.52494 (3)	0.90237 (6)	0.0192 (2)
H2	0.721498	0.534351	0.931197	0.023*
C3	0.58344 (12)	0.50146 (3)	0.93049 (6)	0.0194 (2)
H3	0.610599	0.494813	0.977939	0.023*
C4	0.47584 (12)	0.48781 (3)	0.88817 (7)	0.0203 (2)
H4	0.430003	0.472469	0.907717	0.024*
C5	0.43666 (11)	0.49703 (3)	0.81683 (6)	0.0176 (2)
H5	0.364754	0.487840	0.788474	0.021*
C6	0.50568 (11)	0.52020 (3)	0.78760 (6)	0.0136 (2)
C7	0.53838 (12)	0.46861 (3)	0.66991 (7)	0.0198 (2)
H7	0.588550	0.469321	0.716034	0.024*
C8	0.54019 (13)	0.44169 (3)	0.62722 (8)	0.0254 (3)
H8	0.592086	0.424523	0.644795	0.030*
C9	0.46540 (14)	0.44035 (3)	0.55898 (8)	0.0306 (3)
H9	0.466881	0.422331	0.530569	0.037*
C10	0.38826 (17)	0.46583 (4)	0.53291 (8)	0.0403 (4)
H10	0.337265	0.464822	0.487039	0.048*
C11	0.38629 (15)	0.49291 (4)	0.57465 (7)	0.0312 (3)
H11	0.334659	0.510043	0.556555	0.037*
C12	0.46162 (11)	0.49447 (3)	0.64372 (6)	0.0160 (2)
C13	0.68681 (11)	0.54783 (3)	0.66017 (6)	0.0168 (2)
H13	0.710047	0.526522	0.666794	0.020*
C14	0.77099 (12)	0.56959 (3)	0.63990 (6)	0.0203 (2)

H14	0.850763	0.562941	0.633391	0.024*
C15	0.73532 (12)	0.60136 (3)	0.62942 (7)	0.0223 (3)
H15	0.790696	0.615862	0.614766	0.027*
C16	0.61775 (13)	0.61152 (3)	0.64067 (7)	0.0228 (3)
H16	0.595084	0.632870	0.634256	0.027*
C17	0.53354 (12)	0.58992 (3)	0.66151 (6)	0.0187 (2)
H17	0.454903	0.596820	0.669357	0.022*
C18	0.56734 (11)	0.55785 (3)	0.67064 (6)	0.0141 (2)
P2	−0.04151 (3)	0.53337 (2)	0.18925 (2)	0.01230 (6)
O2	−0.17363 (8)	0.54704 (2)	0.17137 (4)	0.01732 (17)
C19	0.11406 (11)	0.53774 (3)	0.32684 (6)	0.0167 (2)
H19	0.160010	0.553259	0.307909	0.020*
C20	0.15104 (12)	0.52863 (3)	0.39889 (6)	0.0194 (2)
H20	0.221743	0.538005	0.427976	0.023*
C21	0.08202 (12)	0.50557 (3)	0.42697 (6)	0.0205 (2)
H21	0.107516	0.499240	0.474765	0.025*
C22	−0.02503 (12)	0.49184 (3)	0.38417 (7)	0.0211 (2)
H22	−0.071898	0.476702	0.403634	0.025*
C23	−0.06198 (11)	0.50071 (3)	0.31239 (6)	0.0176 (2)
H23	−0.133366	0.491457	0.283687	0.021*
C24	0.00844 (11)	0.52362 (3)	0.28324 (6)	0.0139 (2)
C25	0.04771 (12)	0.47170 (3)	0.16729 (6)	0.0187 (2)
H25	0.099716	0.473162	0.212801	0.022*
C26	0.04960 (12)	0.44426 (3)	0.12632 (7)	0.0226 (3)
H26	0.103258	0.427486	0.144451	0.027*
C27	−0.02776 (13)	0.44180 (3)	0.05889 (7)	0.0257 (3)
H27	−0.025983	0.423458	0.031542	0.031*
C28	−0.10793 (15)	0.46669 (4)	0.03213 (8)	0.0345 (3)
H28	−0.160726	0.464959	−0.013102	0.041*
C29	−0.11000 (14)	0.49422 (3)	0.07241 (7)	0.0280 (3)
H29	−0.163834	0.510912	0.053961	0.034*
C30	−0.03175 (11)	0.49697 (3)	0.14038 (6)	0.0151 (2)
C31	0.03988 (12)	0.59295 (3)	0.15930 (7)	0.0192 (2)
H31	−0.039378	0.599663	0.166545	0.023*
C32	0.12523 (13)	0.61484 (3)	0.14071 (7)	0.0234 (3)
H32	0.102725	0.636236	0.135335	0.028*
C33	0.24365 (12)	0.60499 (3)	0.13014 (7)	0.0212 (3)
H33	0.299701	0.619741	0.117004	0.025*
C34	0.27898 (11)	0.57320 (3)	0.13908 (6)	0.0193 (2)
H34	0.359108	0.566710	0.132823	0.023*
C35	0.19411 (11)	0.55108 (3)	0.15742 (6)	0.0161 (2)
H35	0.217465	0.529756	0.163287	0.019*
C36	0.07374 (11)	0.56081 (3)	0.16705 (6)	0.0142 (2)
P3	0.09805 (3)	0.71259 (2)	0.12227 (2)	0.01599 (7)
O3	0.17981 (8)	0.69166 (2)	0.08605 (5)	0.02155 (18)
C37	−0.08417 (12)	0.68424 (3)	0.19179 (7)	0.0244 (3)
H37	−0.030988	0.688982	0.235659	0.029*
C38	−0.19660 (13)	0.66742 (3)	0.19036 (8)	0.0315 (3)

H38	−0.218007	0.660839	0.233340	0.038*
C39	−0.27655 (13)	0.66046 (3)	0.12557 (9)	0.0312 (3)
H39	−0.351472	0.649149	0.124875	0.037*
C40	−0.24498 (14)	0.67032 (3)	0.06181 (8)	0.0323 (3)
H40	−0.299242	0.665798	0.018164	0.039*
C41	−0.13297 (14)	0.68691 (3)	0.06245 (7)	0.0274 (3)
H41	−0.112158	0.693373	0.019234	0.033*
C42	−0.05124 (12)	0.69397 (3)	0.12764 (7)	0.0185 (2)
C43	−0.03715 (12)	0.76945 (3)	0.08915 (8)	0.0248 (3)
H43	−0.081000	0.764003	0.125165	0.030*
C44	−0.06608 (13)	0.79759 (3)	0.05104 (8)	0.0299 (3)
H44	−0.128479	0.811110	0.061853	0.036*
C45	−0.00170 (14)	0.80552 (3)	−0.00327 (8)	0.0316 (3)
H45	−0.021237	0.824361	−0.028957	0.038*
C46	0.09146 (14)	0.78551 (3)	−0.01936 (8)	0.0305 (3)
H46	0.134234	0.790946	−0.055853	0.037*
C47	0.12140 (12)	0.75730 (3)	0.01881 (7)	0.0239 (3)
H47	0.183816	0.743850	0.007752	0.029*
C48	0.05757 (12)	0.74923 (3)	0.07371 (7)	0.0193 (2)
C49	0.29856 (12)	0.70791 (3)	0.23785 (7)	0.0233 (3)
H49	0.334641	0.694746	0.207831	0.028*
C50	0.36378 (13)	0.71402 (3)	0.30779 (8)	0.0293 (3)
H50	0.443912	0.705170	0.324258	0.035*
C51	0.30930 (14)	0.73330 (3)	0.35291 (8)	0.0312 (3)
H51	0.352266	0.736994	0.399933	0.037*
C52	0.19116 (14)	0.74706 (3)	0.32820 (8)	0.0308 (3)
H52	0.155341	0.760138	0.358497	0.037*
C53	0.12620 (13)	0.74136 (3)	0.25837 (7)	0.0253 (3)
H53	0.047151	0.750767	0.241795	0.030*
C54	0.17919 (12)	0.72151 (3)	0.21276 (7)	0.0188 (2)
P4	0.59434 (3)	0.71241 (2)	0.62054 (2)	0.01558 (7)
O4	0.65852 (8)	0.68597 (2)	0.58832 (4)	0.01947 (18)
C55	0.36994 (13)	0.67928 (3)	0.59742 (8)	0.0282 (3)
H55	0.389594	0.674863	0.552729	0.034*
C56	0.26067 (15)	0.66662 (4)	0.61513 (9)	0.0361 (3)
H56	0.206716	0.653874	0.582208	0.043*
C57	0.23188 (14)	0.67296 (4)	0.68190 (9)	0.0357 (3)
H57	0.158804	0.664312	0.693791	0.043*
C58	0.31085 (14)	0.69200 (4)	0.73083 (8)	0.0324 (3)
H58	0.290945	0.696173	0.775564	0.039*
C59	0.42035 (13)	0.70500 (3)	0.71334 (7)	0.0248 (3)
H59	0.473371	0.717929	0.746290	0.030*
C60	0.45058 (12)	0.69863 (3)	0.64644 (7)	0.0190 (2)
C61	0.77928 (13)	0.70565 (3)	0.74264 (7)	0.0256 (3)
H61	0.789190	0.685356	0.724829	0.031*
C62	0.85001 (14)	0.71467 (4)	0.80939 (8)	0.0328 (3)
H62	0.907827	0.700498	0.835864	0.039*
C63	0.83457 (14)	0.74466 (4)	0.83647 (7)	0.0310 (3)

H63	0.880548	0.750466	0.881635	0.037*
C64	0.75106 (15)	0.76599 (4)	0.79662 (8)	0.0351 (3)
H64	0.741536	0.786242	0.814744	0.042*
C65	0.68114 (14)	0.75731 (3)	0.72948 (8)	0.0313 (3)
H65	0.625732	0.771861	0.702523	0.038*
C66	0.69371 (11)	0.72688 (3)	0.70238 (7)	0.0191 (2)
C67	0.45982 (12)	0.76745 (3)	0.56674 (7)	0.0231 (3)
H67	0.417547	0.765712	0.604988	0.028*
C68	0.43043 (13)	0.79226 (3)	0.51757 (8)	0.0275 (3)
H68	0.369387	0.807282	0.523296	0.033*
C69	0.49220 (13)	0.79458 (3)	0.46002 (8)	0.0283 (3)
H69	0.472196	0.811139	0.427055	0.034*
C70	0.58383 (13)	0.77230 (3)	0.45135 (7)	0.0261 (3)
H70	0.624512	0.773921	0.412430	0.031*
C71	0.61497 (12)	0.74760 (3)	0.50053 (7)	0.0207 (2)
H71	0.676790	0.732791	0.494812	0.025*
C72	0.55292 (11)	0.74510 (3)	0.55873 (7)	0.0183 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01503 (14)	0.02690 (16)	0.01590 (14)	0.00414 (11)	0.00267 (11)	0.00504 (11)
O5	0.1541 (15)	0.0195 (5)	0.0287 (6)	−0.0136 (7)	0.0334 (8)	−0.0030 (4)
O6	0.0519 (6)	0.0196 (5)	0.0228 (5)	−0.0019 (4)	0.0189 (4)	−0.0022 (4)
O7	0.0198 (4)	0.0312 (5)	0.0197 (4)	0.0043 (4)	0.0047 (3)	0.0069 (4)
O8	0.0187 (4)	0.0351 (5)	0.0200 (4)	0.0091 (4)	0.0011 (3)	0.0089 (4)
C73	0.0162 (5)	0.0211 (6)	0.0188 (6)	0.0022 (5)	0.0022 (4)	0.0008 (5)
C74	0.0175 (6)	0.0274 (6)	0.0164 (6)	−0.0040 (5)	0.0004 (4)	−0.0018 (5)
C75	0.0251 (6)	0.0255 (6)	0.0194 (6)	−0.0024 (5)	0.0065 (5)	−0.0073 (5)
C76	0.0211 (6)	0.0232 (6)	0.0249 (6)	0.0056 (5)	0.0072 (5)	−0.0028 (5)
C77	0.0162 (5)	0.0206 (6)	0.0170 (5)	0.0017 (5)	0.0028 (4)	0.0014 (5)
C78	0.0160 (5)	0.0161 (5)	0.0164 (5)	0.0000 (4)	0.0037 (4)	0.0006 (4)
C79	0.0234 (6)	0.0185 (6)	0.0165 (6)	0.0024 (5)	0.0018 (5)	0.0007 (5)
C80	0.0186 (6)	0.0154 (5)	0.0164 (5)	0.0031 (4)	0.0042 (4)	0.0003 (4)
C81	0.0235 (6)	0.0277 (7)	0.0192 (6)	0.0062 (5)	0.0046 (5)	0.0069 (5)
C82	0.0310 (7)	0.0344 (7)	0.0262 (7)	0.0060 (6)	0.0116 (6)	0.0128 (6)
C83	0.0246 (7)	0.0337 (7)	0.0334 (7)	0.0006 (6)	0.0139 (6)	0.0093 (6)
C84	0.0177 (6)	0.0260 (6)	0.0260 (6)	0.0022 (5)	0.0044 (5)	0.0043 (5)
C85	0.0190 (6)	0.0149 (5)	0.0165 (5)	0.0018 (4)	0.0037 (4)	0.0003 (4)
C86	0.0165 (5)	0.0161 (5)	0.0168 (5)	0.0009 (4)	−0.0005 (4)	−0.0003 (4)
S2	0.01312 (13)	0.02878 (16)	0.01450 (13)	0.00328 (11)	0.00294 (10)	0.00385 (11)
O9	0.0180 (4)	0.0253 (4)	0.0169 (4)	0.0024 (3)	0.0033 (3)	0.0036 (3)
O10	0.0172 (4)	0.0317 (5)	0.0182 (4)	0.0088 (4)	0.0007 (3)	0.0046 (4)
O11	0.0794 (9)	0.0169 (5)	0.0236 (5)	−0.0017 (5)	−0.0069 (5)	0.0018 (4)
O12	0.0420 (6)	0.0184 (4)	0.0189 (4)	0.0065 (4)	0.0136 (4)	0.0013 (4)
C87	0.0184 (6)	0.0192 (6)	0.0168 (5)	0.0033 (5)	0.0016 (4)	0.0022 (4)
C88	0.0278 (7)	0.0233 (6)	0.0196 (6)	0.0017 (5)	0.0083 (5)	0.0060 (5)
C89	0.0212 (6)	0.0294 (7)	0.0281 (7)	0.0002 (5)	0.0118 (5)	0.0054 (5)

C90	0.0159 (6)	0.0238 (6)	0.0246 (6)	0.0019 (5)	0.0050 (5)	0.0021 (5)
C91	0.0152 (5)	0.0150 (5)	0.0157 (5)	0.0013 (4)	0.0027 (4)	−0.0007 (4)
C92	0.0166 (5)	0.0137 (5)	0.0152 (5)	0.0014 (4)	0.0037 (4)	−0.0008 (4)
C93	0.0154 (5)	0.0150 (5)	0.0162 (5)	0.0005 (4)	−0.0003 (4)	−0.0012 (4)
C94	0.0138 (5)	0.0237 (6)	0.0167 (5)	−0.0001 (5)	0.0037 (4)	0.0023 (5)
C95	0.0187 (6)	0.0264 (6)	0.0236 (6)	0.0027 (5)	0.0073 (5)	−0.0017 (5)
C96	0.0227 (6)	0.0292 (7)	0.0193 (6)	−0.0042 (5)	0.0063 (5)	−0.0050 (5)
C97	0.0177 (6)	0.0309 (7)	0.0167 (6)	−0.0058 (5)	−0.0003 (5)	−0.0003 (5)
C98	0.0151 (5)	0.0234 (6)	0.0193 (6)	−0.0011 (5)	0.0024 (5)	0.0035 (5)
C99	0.0142 (5)	0.0186 (6)	0.0166 (5)	−0.0027 (4)	0.0036 (4)	0.0027 (4)
C100	0.0155 (5)	0.0191 (6)	0.0179 (6)	−0.0015 (4)	−0.0007 (4)	0.0024 (5)
P1	0.01150 (13)	0.01351 (13)	0.01110 (13)	0.00026 (10)	0.00034 (10)	0.00070 (10)
O1	0.0128 (4)	0.0217 (4)	0.0176 (4)	0.0025 (3)	0.0013 (3)	0.0031 (3)
C1	0.0172 (6)	0.0182 (5)	0.0144 (5)	−0.0027 (4)	0.0023 (4)	−0.0004 (4)
C2	0.0172 (6)	0.0259 (6)	0.0133 (5)	−0.0021 (5)	−0.0001 (4)	−0.0022 (5)
C3	0.0210 (6)	0.0254 (6)	0.0120 (5)	0.0037 (5)	0.0035 (4)	0.0022 (5)
C4	0.0228 (6)	0.0210 (6)	0.0180 (6)	−0.0027 (5)	0.0062 (5)	0.0037 (5)
C5	0.0172 (5)	0.0188 (6)	0.0161 (5)	−0.0027 (5)	0.0014 (4)	−0.0002 (4)
C6	0.0146 (5)	0.0137 (5)	0.0125 (5)	0.0017 (4)	0.0024 (4)	−0.0003 (4)
C7	0.0202 (6)	0.0193 (6)	0.0196 (6)	0.0003 (5)	0.0030 (5)	−0.0023 (5)
C8	0.0257 (7)	0.0180 (6)	0.0349 (7)	−0.0006 (5)	0.0118 (6)	−0.0047 (5)
C9	0.0329 (7)	0.0278 (7)	0.0336 (7)	−0.0083 (6)	0.0124 (6)	−0.0168 (6)
C10	0.0489 (10)	0.0429 (9)	0.0232 (7)	0.0014 (7)	−0.0069 (7)	−0.0169 (6)
C11	0.0382 (8)	0.0311 (7)	0.0190 (6)	0.0060 (6)	−0.0069 (6)	−0.0056 (5)
C12	0.0155 (5)	0.0173 (5)	0.0149 (5)	−0.0032 (4)	0.0026 (4)	−0.0016 (4)
C13	0.0167 (5)	0.0175 (5)	0.0153 (5)	0.0000 (4)	0.0015 (4)	−0.0014 (4)
C14	0.0156 (5)	0.0280 (6)	0.0172 (6)	−0.0037 (5)	0.0032 (4)	−0.0022 (5)
C15	0.0227 (6)	0.0253 (6)	0.0175 (6)	−0.0089 (5)	0.0009 (5)	0.0035 (5)
C16	0.0272 (7)	0.0166 (6)	0.0232 (6)	−0.0022 (5)	0.0016 (5)	0.0043 (5)
C17	0.0186 (6)	0.0174 (6)	0.0195 (6)	0.0016 (5)	0.0024 (5)	0.0015 (4)
C18	0.0155 (5)	0.0162 (5)	0.0096 (5)	−0.0013 (4)	0.0000 (4)	0.0002 (4)
P2	0.01140 (13)	0.01358 (13)	0.01122 (13)	0.00096 (10)	0.00054 (10)	0.00039 (10)
O2	0.0131 (4)	0.0211 (4)	0.0170 (4)	0.0036 (3)	0.0013 (3)	0.0014 (3)
C19	0.0175 (6)	0.0175 (5)	0.0151 (5)	−0.0020 (4)	0.0028 (4)	−0.0006 (4)
C20	0.0178 (6)	0.0253 (6)	0.0136 (5)	−0.0021 (5)	−0.0002 (4)	−0.0030 (5)
C21	0.0222 (6)	0.0265 (6)	0.0126 (5)	0.0023 (5)	0.0031 (5)	0.0018 (5)
C22	0.0235 (6)	0.0232 (6)	0.0171 (6)	−0.0031 (5)	0.0058 (5)	0.0032 (5)
C23	0.0169 (5)	0.0193 (6)	0.0159 (5)	−0.0023 (5)	0.0019 (4)	−0.0003 (4)
C24	0.0148 (5)	0.0140 (5)	0.0129 (5)	0.0023 (4)	0.0027 (4)	−0.0005 (4)
C25	0.0184 (6)	0.0192 (6)	0.0176 (6)	0.0011 (5)	0.0013 (5)	−0.0015 (5)
C26	0.0228 (6)	0.0175 (6)	0.0288 (7)	0.0017 (5)	0.0085 (5)	−0.0025 (5)
C27	0.0255 (7)	0.0243 (6)	0.0288 (7)	−0.0048 (5)	0.0093 (5)	−0.0125 (5)
C28	0.0375 (8)	0.0377 (8)	0.0231 (7)	0.0042 (7)	−0.0066 (6)	−0.0143 (6)
C29	0.0323 (7)	0.0288 (7)	0.0183 (6)	0.0086 (6)	−0.0062 (5)	−0.0050 (5)
C30	0.0142 (5)	0.0166 (5)	0.0146 (5)	−0.0010 (4)	0.0030 (4)	−0.0014 (4)
C31	0.0189 (6)	0.0175 (6)	0.0215 (6)	0.0031 (5)	0.0046 (5)	0.0019 (5)
C32	0.0275 (7)	0.0154 (6)	0.0273 (6)	0.0013 (5)	0.0051 (5)	0.0048 (5)
C33	0.0218 (6)	0.0221 (6)	0.0189 (6)	−0.0056 (5)	0.0020 (5)	0.0043 (5)

C34	0.0155 (5)	0.0247 (6)	0.0176 (6)	−0.0018 (5)	0.0027 (4)	−0.0016 (5)
C35	0.0169 (6)	0.0154 (5)	0.0153 (5)	0.0010 (4)	0.0012 (4)	−0.0010 (4)
C36	0.0157 (5)	0.0158 (5)	0.0101 (5)	0.0001 (4)	0.0002 (4)	−0.0002 (4)
P3	0.01686 (14)	0.01392 (14)	0.01790 (14)	0.00187 (11)	0.00514 (11)	−0.00059 (11)
O3	0.0249 (4)	0.0197 (4)	0.0215 (4)	0.0058 (4)	0.0082 (4)	−0.0020 (3)
C37	0.0198 (6)	0.0265 (6)	0.0257 (6)	−0.0007 (5)	0.0020 (5)	0.0058 (5)
C38	0.0247 (7)	0.0321 (7)	0.0380 (8)	−0.0025 (6)	0.0066 (6)	0.0114 (6)
C39	0.0210 (6)	0.0197 (6)	0.0507 (9)	−0.0035 (5)	0.0018 (6)	0.0018 (6)
C40	0.0296 (7)	0.0229 (7)	0.0390 (8)	−0.0050 (6)	−0.0060 (6)	−0.0052 (6)
C41	0.0332 (7)	0.0230 (6)	0.0245 (6)	−0.0046 (6)	0.0020 (5)	−0.0029 (5)
C42	0.0184 (6)	0.0124 (5)	0.0241 (6)	0.0016 (4)	0.0032 (5)	−0.0001 (4)
C43	0.0209 (6)	0.0217 (6)	0.0337 (7)	0.0023 (5)	0.0102 (5)	0.0037 (5)
C44	0.0231 (6)	0.0202 (6)	0.0472 (8)	0.0053 (5)	0.0085 (6)	0.0047 (6)
C45	0.0305 (7)	0.0203 (6)	0.0432 (8)	0.0004 (6)	0.0054 (6)	0.0114 (6)
C46	0.0315 (7)	0.0281 (7)	0.0339 (7)	−0.0005 (6)	0.0109 (6)	0.0091 (6)
C47	0.0231 (6)	0.0227 (6)	0.0273 (6)	0.0015 (5)	0.0083 (5)	0.0032 (5)
C48	0.0182 (6)	0.0162 (6)	0.0234 (6)	−0.0002 (5)	0.0039 (5)	0.0014 (5)
C49	0.0202 (6)	0.0230 (6)	0.0270 (7)	0.0017 (5)	0.0051 (5)	−0.0018 (5)
C50	0.0215 (6)	0.0308 (7)	0.0325 (7)	0.0011 (5)	−0.0021 (5)	0.0002 (6)
C51	0.0353 (8)	0.0301 (7)	0.0249 (7)	−0.0059 (6)	−0.0019 (6)	−0.0052 (6)
C52	0.0379 (8)	0.0267 (7)	0.0279 (7)	0.0008 (6)	0.0065 (6)	−0.0095 (6)
C53	0.0257 (6)	0.0234 (6)	0.0266 (7)	0.0039 (5)	0.0046 (5)	−0.0051 (5)
C54	0.0185 (6)	0.0165 (5)	0.0215 (6)	−0.0015 (5)	0.0040 (5)	−0.0014 (5)
P4	0.01651 (14)	0.01290 (14)	0.01749 (14)	0.00079 (11)	0.00375 (11)	−0.00068 (11)
O4	0.0241 (4)	0.0156 (4)	0.0196 (4)	0.0037 (3)	0.0063 (3)	−0.0012 (3)
C55	0.0279 (7)	0.0244 (7)	0.0328 (7)	−0.0063 (5)	0.0073 (6)	−0.0046 (5)
C56	0.0295 (7)	0.0259 (7)	0.0530 (9)	−0.0089 (6)	0.0084 (7)	−0.0013 (7)
C57	0.0253 (7)	0.0294 (7)	0.0562 (10)	0.0005 (6)	0.0170 (7)	0.0153 (7)
C58	0.0294 (7)	0.0373 (8)	0.0344 (7)	0.0095 (6)	0.0157 (6)	0.0123 (6)
C59	0.0236 (6)	0.0267 (7)	0.0251 (6)	0.0043 (5)	0.0071 (5)	0.0026 (5)
C60	0.0193 (6)	0.0143 (5)	0.0239 (6)	0.0025 (5)	0.0055 (5)	0.0036 (5)
C61	0.0236 (6)	0.0248 (6)	0.0268 (7)	0.0015 (5)	0.0015 (5)	−0.0020 (5)
C62	0.0287 (7)	0.0372 (8)	0.0288 (7)	0.0001 (6)	−0.0033 (6)	0.0010 (6)
C63	0.0282 (7)	0.0435 (8)	0.0216 (6)	−0.0135 (6)	0.0058 (5)	−0.0076 (6)
C64	0.0381 (8)	0.0325 (8)	0.0342 (8)	−0.0052 (6)	0.0061 (6)	−0.0163 (6)
C65	0.0340 (7)	0.0241 (7)	0.0330 (7)	0.0036 (6)	0.0001 (6)	−0.0085 (6)
C66	0.0173 (6)	0.0205 (6)	0.0202 (6)	−0.0024 (5)	0.0050 (5)	−0.0025 (5)
C67	0.0192 (6)	0.0202 (6)	0.0309 (7)	0.0021 (5)	0.0072 (5)	0.0029 (5)
C68	0.0197 (6)	0.0213 (6)	0.0414 (8)	0.0045 (5)	0.0056 (6)	0.0067 (6)
C69	0.0258 (7)	0.0227 (6)	0.0352 (7)	0.0012 (5)	0.0028 (6)	0.0117 (6)
C70	0.0260 (7)	0.0248 (6)	0.0282 (7)	−0.0024 (5)	0.0074 (5)	0.0056 (5)
C71	0.0189 (6)	0.0181 (6)	0.0255 (6)	−0.0010 (5)	0.0052 (5)	0.0006 (5)
C72	0.0164 (6)	0.0153 (5)	0.0226 (6)	−0.0020 (4)	0.0024 (5)	0.0009 (5)

Geometric parameters (Å, º)

S1—C77	1.7839 (12)	C20—H20	0.9300
S1—C80	1.7764 (12)	C20—C21	1.3866 (18)

O5—C79	1.1978 (17)	C21—H21	0.9300
O6—H6O	0.95 (2)	C21—C22	1.3902 (18)
O6—C79	1.3043 (15)	C22—H22	0.9300
O7—C86	1.2104 (15)	C22—C23	1.3880 (17)
O8—H8O	0.90 (2)	C23—H23	0.9300
O8—C86	1.3279 (15)	C23—C24	1.4015 (16)
C73—H73	0.9300	C25—H25	0.9300
C73—C74	1.3862 (17)	C25—C26	1.3910 (17)
C73—C78	1.3942 (17)	C25—C30	1.3914 (17)
C74—H74	0.9300	C26—H26	0.9300
C74—C75	1.3866 (19)	C26—C27	1.3799 (19)
C75—H75	0.9300	C27—H27	0.9300
C75—C76	1.3854 (19)	C27—C28	1.383 (2)
C76—H76	0.9300	C28—H28	0.9300
C76—C77	1.3975 (17)	C28—C29	1.3871 (19)
C77—C78	1.3996 (16)	C29—H29	0.9300
C78—C79	1.4998 (16)	C29—C30	1.3933 (17)
C80—C81	1.4024 (16)	C31—H31	0.9300
C80—C85	1.4077 (17)	C31—C32	1.3898 (18)
C81—H81	0.9300	C31—C36	1.3969 (16)
C81—C82	1.3847 (19)	C32—H32	0.9300
C82—H82	0.9300	C32—C33	1.3857 (19)
C82—C83	1.388 (2)	C33—H33	0.9300
C83—H83	0.9300	C33—C34	1.3883 (18)
C83—C84	1.3826 (18)	C34—H34	0.9300
C84—H84	0.9300	C34—C35	1.3907 (17)
C84—C85	1.4004 (17)	C35—H35	0.9300
C85—C86	1.4921 (16)	C35—C36	1.3983 (16)
S2—C92	1.7760 (12)	P3—O3	1.4991 (9)
S2—C94	1.7863 (12)	P3—C42	1.8004 (13)
O9—C93	1.2147 (15)	P3—C48	1.8001 (12)
O10—H10O	0.91 (2)	P3—C54	1.7989 (13)
O10—C93	1.3309 (14)	C37—H37	0.9300
O11—C100	1.2075 (16)	C37—C38	1.3912 (19)
O12—H12O	0.89 (2)	C37—C42	1.3915 (17)
O12—C100	1.3126 (15)	C38—H38	0.9300
C87—H87	0.9300	C38—C39	1.380 (2)
C87—C88	1.3842 (17)	C39—H39	0.9300
C87—C92	1.4042 (16)	C39—C40	1.380 (2)
C88—H88	0.9300	C40—H40	0.9300
C88—C89	1.3887 (19)	C40—C41	1.385 (2)
C89—H89	0.9300	C41—H41	0.9300
C89—C90	1.3853 (18)	C41—C42	1.3949 (18)
C90—H90	0.9300	C43—H43	0.9300
C90—C91	1.4008 (16)	C43—C44	1.3865 (19)
C91—C92	1.4087 (16)	C43—C48	1.3973 (17)
C91—C93	1.4900 (16)	C44—H44	0.9300
C94—C95	1.3980 (17)	C44—C45	1.388 (2)

C94—C99	1.4031 (17)	C45—H45	0.9300
C95—H95	0.9300	C45—C46	1.384 (2)
C95—C96	1.3879 (18)	C46—H46	0.9300
C96—H96	0.9300	C46—C47	1.3911 (18)
C96—C97	1.3876 (19)	C47—H47	0.9300
C97—H97	0.9300	C47—C48	1.3943 (17)
C97—C98	1.3856 (18)	C49—H49	0.9300
C98—H98	0.9300	C49—C50	1.3919 (19)
C98—C99	1.3979 (16)	C49—C54	1.3941 (18)
C99—C100	1.4997 (17)	C50—H50	0.9300
P1—O1	1.5018 (8)	C50—C51	1.387 (2)
P1—C6	1.7961 (11)	C51—H51	0.9300
P1—C12	1.8003 (12)	C51—C52	1.385 (2)
P1—C18	1.7994 (12)	C52—H52	0.9300
C1—H1	0.9300	C52—C53	1.3863 (19)
C1—C2	1.3952 (16)	C53—H53	0.9300
C1—C6	1.3909 (16)	C53—C54	1.3987 (17)
C2—H2	0.9300	P4—O4	1.4975 (8)
C2—C3	1.3874 (18)	P4—C60	1.8014 (13)
C3—H3	0.9300	P4—C66	1.8010 (13)
C3—C4	1.3890 (18)	P4—C72	1.8005 (12)
C4—H4	0.9300	C55—H55	0.9300
C4—C5	1.3868 (17)	C55—C56	1.386 (2)
C5—H5	0.9300	C55—C60	1.3955 (18)
C5—C6	1.4007 (16)	C56—H56	0.9300
C7—H7	0.9300	C56—C57	1.385 (2)
C7—C8	1.3915 (17)	C57—H57	0.9300
C7—C12	1.3920 (17)	C57—C58	1.378 (2)
C8—H8	0.9300	C58—H58	0.9300
C8—C9	1.379 (2)	C58—C59	1.3917 (19)
C9—H9	0.9300	C59—H59	0.9300
C9—C10	1.381 (2)	C59—C60	1.3937 (18)
C10—H10	0.9300	C61—H61	0.9300
C10—C11	1.387 (2)	C61—C62	1.3899 (19)
C11—H11	0.9300	C61—C66	1.3915 (18)
C11—C12	1.3941 (17)	C62—H62	0.9300
C13—H13	0.9300	C62—C63	1.381 (2)
C13—C14	1.3901 (17)	C63—H63	0.9300
C13—C18	1.3979 (16)	C63—C64	1.379 (2)
C14—H14	0.9300	C64—H64	0.9300
C14—C15	1.3902 (18)	C64—C65	1.389 (2)
C15—H15	0.9300	C65—H65	0.9300
C15—C16	1.3854 (19)	C65—C66	1.3929 (18)
C16—H16	0.9300	C67—H67	0.9300
C16—C17	1.3902 (17)	C67—C68	1.3902 (18)
C17—H17	0.9300	C67—C72	1.3991 (17)
C17—C18	1.3956 (16)	C68—H68	0.9300
P2—O2	1.5014 (8)	C68—C69	1.386 (2)

P2—C24	1.7997 (11)	C69—H69	0.9300
P2—C30	1.7998 (12)	C69—C70	1.389 (2)
P2—C36	1.7980 (12)	C70—H70	0.9300
C19—H19	0.9300	C70—C71	1.3889 (18)
C19—C20	1.3956 (16)	C71—H71	0.9300
C19—C24	1.3927 (16)	C71—C72	1.3990 (17)

C80—S1—C77	101.78 (6)	C21—C22—H22	120.0
C79—O6—H6O	109.8 (14)	C23—C22—C21	120.02 (11)
C86—O8—H8O	111.7 (14)	C23—C22—H22	120.0
C74—C73—H73	119.7	C22—C23—H23	120.0
C74—C73—C78	120.70 (11)	C22—C23—C24	119.91 (11)
C78—C73—H73	119.7	C24—C23—H23	120.0
C73—C74—H74	120.2	C19—C24—P2	122.36 (9)
C73—C74—C75	119.62 (11)	C19—C24—C23	119.73 (10)
C75—C74—H74	120.2	C23—C24—P2	117.90 (9)
C74—C75—H75	119.9	C26—C25—H25	119.9
C76—C75—C74	120.21 (11)	C26—C25—C30	120.21 (11)
C76—C75—H75	119.9	C30—C25—H25	119.9
C75—C76—H76	119.6	C25—C26—H26	119.8
C75—C76—C77	120.71 (12)	C27—C26—C25	120.31 (12)
C77—C76—H76	119.6	C27—C26—H26	119.8
C76—C77—S1	119.72 (9)	C26—C27—H27	120.1
C76—C77—C78	118.98 (11)	C26—C27—C28	119.79 (12)
C78—C77—S1	121.28 (9)	C28—C27—H27	120.1
C73—C78—C77	119.77 (11)	C27—C28—H28	119.8
C73—C78—C79	116.79 (11)	C27—C28—C29	120.33 (13)
C77—C78—C79	123.40 (11)	C29—C28—H28	119.8
O5—C79—O6	123.45 (12)	C28—C29—H29	119.9
O5—C79—C78	121.67 (12)	C28—C29—C30	120.27 (13)
O6—C79—C78	114.76 (11)	C30—C29—H29	119.9
C81—C80—S1	122.19 (9)	C25—C30—P2	123.58 (9)
C81—C80—C85	117.98 (11)	C25—C30—C29	119.09 (11)
C85—C80—S1	119.81 (9)	C29—C30—P2	117.33 (9)
C80—C81—H81	119.5	C32—C31—H31	120.2
C82—C81—C80	121.10 (12)	C32—C31—C36	119.67 (11)
C82—C81—H81	119.5	C36—C31—H31	120.2
C81—C82—H82	119.6	C31—C32—H32	119.8
C81—C82—C83	120.85 (12)	C33—C32—C31	120.40 (12)
C83—C82—H82	119.6	C33—C32—H32	119.8
C82—C83—H83	120.6	C32—C33—H33	119.9
C84—C83—C82	118.84 (12)	C32—C33—C34	120.29 (11)
C84—C83—H83	120.6	C34—C33—H33	119.9
C83—C84—H84	119.4	C33—C34—H34	120.1
C83—C84—C85	121.26 (12)	C33—C34—C35	119.76 (11)
C85—C84—H84	119.4	C35—C34—H34	120.1
C80—C85—C86	121.64 (11)	C34—C35—H35	119.9
C84—C85—C80	119.94 (11)	C34—C35—C36	120.18 (11)

C84—C85—C86	118.41 (11)	C36—C35—H35	119.9
O7—C86—O8	124.24 (11)	C31—C36—P2	117.85 (9)
O7—C86—C85	123.93 (11)	C31—C36—C35	119.68 (11)
O8—C86—C85	111.83 (10)	C35—C36—P2	122.46 (9)
C92—S2—C94	100.52 (5)	O3—P3—C42	112.18 (5)
C93—O10—H10O	110.6 (14)	O3—P3—C48	111.75 (5)
C100—O12—H12O	111.0 (12)	O3—P3—C54	110.01 (6)
C88—C87—H87	119.4	C48—P3—C42	105.87 (6)
C88—C87—C92	121.16 (11)	C54—P3—C42	107.61 (6)
C92—C87—H87	119.4	C54—P3—C48	109.24 (6)
C87—C88—H88	119.7	C38—C37—H37	120.0
C87—C88—C89	120.59 (11)	C38—C37—C42	120.05 (13)
C89—C88—H88	119.7	C42—C37—H37	120.0
C88—C89—H89	120.5	C37—C38—H38	119.8
C90—C89—C88	119.06 (12)	C39—C38—C37	120.41 (13)
C90—C89—H89	120.5	C39—C38—H38	119.8
C89—C90—H90	119.4	C38—C39—H39	120.1
C89—C90—C91	121.30 (12)	C38—C39—C40	119.79 (13)
C91—C90—H90	119.4	C40—C39—H39	120.1
C90—C91—C92	119.64 (11)	C39—C40—H40	119.8
C90—C91—C93	118.82 (11)	C39—C40—C41	120.39 (13)
C92—C91—C93	121.50 (10)	C41—C40—H40	119.8
C87—C92—S2	121.33 (9)	C40—C41—H41	119.9
C87—C92—C91	118.24 (11)	C40—C41—C42	120.28 (13)
C91—C92—S2	120.40 (9)	C42—C41—H41	119.9
O9—C93—O10	123.83 (11)	C37—C42—P3	123.98 (10)
O9—C93—C91	123.56 (11)	C37—C42—C41	119.08 (12)
O10—C93—C91	112.61 (10)	C41—C42—P3	116.74 (10)
C95—C94—S2	118.49 (9)	C44—C43—H43	119.8
C95—C94—C99	119.21 (11)	C44—C43—C48	120.32 (12)
C99—C94—S2	122.30 (9)	C48—C43—H43	119.8
C94—C95—H95	119.6	C43—C44—H44	120.1
C96—C95—C94	120.76 (12)	C43—C44—C45	119.82 (13)
C96—C95—H95	119.6	C45—C44—H44	120.1
C95—C96—H96	120.0	C44—C45—H45	119.9
C97—C96—C95	120.05 (12)	C46—C45—C44	120.25 (13)
C97—C96—H96	120.0	C46—C45—H45	119.9
C96—C97—H97	120.1	C45—C46—H46	119.9
C98—C97—C96	119.71 (12)	C45—C46—C47	120.28 (13)
C98—C97—H97	120.1	C47—C46—H46	119.9
C97—C98—H98	119.5	C46—C47—H47	120.1
C97—C98—C99	120.98 (12)	C46—C47—C48	119.82 (12)
C99—C98—H98	119.5	C48—C47—H47	120.1
C94—C99—C100	124.32 (11)	C43—C48—P3	121.55 (10)
C98—C99—C94	119.28 (11)	C47—C48—P3	118.94 (9)
C98—C99—C100	116.38 (11)	C47—C48—C43	119.50 (12)
O11—C100—O12	123.80 (12)	C50—C49—H49	120.0
O11—C100—C99	121.95 (11)	C50—C49—C54	120.05 (12)

O12—C100—C99	114.18 (10)	C54—C49—H49	120.0
O1—P1—C6	112.35 (5)	C49—C50—H50	120.0
O1—P1—C12	111.32 (5)	C51—C50—C49	120.01 (13)
O1—P1—C18	111.73 (5)	C51—C50—H50	120.0
C6—P1—C12	106.40 (5)	C50—C51—H51	119.9
C6—P1—C18	107.57 (5)	C52—C51—C50	120.25 (13)
C18—P1—C12	107.14 (5)	C52—C51—H51	119.9
C2—C1—H1	120.0	C51—C52—H52	120.0
C6—C1—H1	120.0	C51—C52—C53	120.08 (13)
C6—C1—C2	120.03 (11)	C53—C52—H52	120.0
C1—C2—H2	120.1	C52—C53—H53	119.9
C3—C2—C1	119.77 (11)	C52—C53—C54	120.19 (13)
C3—C2—H2	120.1	C54—C53—H53	119.9
C2—C3—H3	119.8	C49—C54—P3	118.37 (9)
C2—C3—C4	120.38 (11)	C49—C54—C53	119.40 (12)
C4—C3—H3	119.8	C53—C54—P3	122.23 (10)
C3—C4—H4	119.9	O4—P4—C60	111.07 (5)
C5—C4—C3	120.12 (11)	O4—P4—C66	111.08 (5)
C5—C4—H4	119.9	O4—P4—C72	111.95 (5)
C4—C5—H5	120.1	C66—P4—C60	105.35 (6)
C4—C5—C6	119.81 (11)	C72—P4—C60	107.95 (6)
C6—C5—H5	120.1	C72—P4—C66	109.19 (6)
C1—C6—P1	122.24 (9)	C56—C55—H55	119.9
C1—C6—C5	119.86 (10)	C56—C55—C60	120.19 (13)
C5—C6—P1	117.90 (9)	C60—C55—H55	119.9
C8—C7—H7	119.9	C55—C56—H56	120.0
C8—C7—C12	120.14 (12)	C57—C56—C55	119.95 (14)
C12—C7—H7	119.9	C57—C56—H56	120.0
C7—C8—H8	119.9	C56—C57—H57	119.8
C9—C8—C7	120.28 (13)	C58—C57—C56	120.41 (13)
C9—C8—H8	119.9	C58—C57—H57	119.8
C8—C9—H9	120.1	C57—C58—H58	120.0
C8—C9—C10	119.85 (12)	C57—C58—C59	120.05 (14)
C10—C9—H9	120.1	C59—C58—H58	120.0
C9—C10—H10	119.8	C58—C59—H59	120.0
C9—C10—C11	120.47 (13)	C58—C59—C60	120.01 (13)
C11—C10—H10	119.8	C60—C59—H59	120.0
C10—C11—H11	120.0	C55—C60—P4	117.07 (10)
C10—C11—C12	120.08 (13)	C59—C60—P4	123.46 (10)
C12—C11—H11	120.0	C59—C60—C55	119.39 (12)
C7—C12—P1	123.65 (9)	C62—C61—H61	119.9
C7—C12—C11	119.17 (11)	C62—C61—C66	120.23 (13)
C11—C12—P1	117.18 (10)	C66—C61—H61	119.9
C14—C13—H13	119.9	C61—C62—H62	119.9
C14—C13—C18	120.22 (11)	C63—C62—C61	120.12 (14)
C18—C13—H13	119.9	C63—C62—H62	119.9
C13—C14—H14	120.2	C62—C63—H63	120.0
C13—C14—C15	119.66 (12)	C64—C63—C62	120.07 (13)

C15—C14—H14	120.2	C64—C63—H63	120.0
C14—C15—H15	119.8	C63—C64—H64	119.9
C16—C15—C14	120.38 (12)	C63—C64—C65	120.19 (13)
C16—C15—H15	119.8	C65—C64—H64	119.9
C15—C16—H16	119.9	C64—C65—H65	119.9
C15—C16—C17	120.22 (12)	C64—C65—C66	120.23 (14)
C17—C16—H16	119.9	C66—C65—H65	119.9
C16—C17—H17	120.1	C61—C66—P4	117.83 (10)
C16—C17—C18	119.82 (11)	C61—C66—C65	119.14 (12)
C18—C17—H17	120.1	C65—C66—P4	122.83 (10)
C13—C18—P1	121.99 (9)	C68—C67—H67	120.0
C17—C18—P1	118.34 (9)	C68—C67—C72	120.06 (12)
C17—C18—C13	119.66 (11)	C72—C67—H67	120.0
O2—P2—C24	113.00 (5)	C67—C68—H68	120.0
O2—P2—C30	110.92 (5)	C69—C68—C67	119.93 (12)
O2—P2—C36	111.25 (5)	C69—C68—H68	120.0
C24—P2—C30	106.18 (5)	C68—C69—H69	119.8
C36—P2—C24	107.09 (5)	C68—C69—C70	120.30 (12)
C36—P2—C30	108.12 (5)	C70—C69—H69	119.8
C20—C19—H19	120.0	C69—C70—H70	119.9
C24—C19—H19	120.0	C69—C70—C71	120.30 (12)
C24—C19—C20	120.09 (11)	C71—C70—H70	119.9
C19—C20—H20	120.1	C70—C71—H71	120.2
C21—C20—C19	119.78 (11)	C70—C71—C72	119.70 (12)
C21—C20—H20	120.1	C72—C71—H71	120.2
C20—C21—H21	119.8	C67—C72—P4	121.96 (10)
C20—C21—C22	120.45 (11)	C67—C72—C71	119.70 (11)
C22—C21—H21	119.8	C71—C72—P4	118.33 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6*O*···O4	0.95 (2)	1.66 (2)	2.6070 (12)	171 (2)
O8—H8*O*···O1^{i}	0.90 (2)	1.70 (2)	2.5763 (12)	163 (2)
O10—H10*O*···O2^{ii}	0.91 (2)	1.72 (2)	2.6077 (12)	163 (2)
O12—H12*O*···O3^{iii}	0.90 (2)	1.71 (2)	2.5978 (12)	170.9 (19)
C16—H16···O4	0.93	2.53	3.3333 (15)	144
C44—H44···O4^{iv}	0.93	2.43	3.2404 (17)	145
C52—H52···O11^{v}	0.93	2.49	3.3231 (16)	149
C62—H62···O11^{i}	0.93	2.51	3.367 (2)	153
C64—H64···O5^{vi}	0.93	2.46	3.263 (2)	144
C68—H68···O3^{vi}	0.93	2.55	3.2747 (17)	135
C71—H71···O5	0.93	2.59	3.2765 (18)	131
C75—H75···O2^{i}	0.93	2.41	3.1184 (16)	133
C96—H96···O1	0.93	2.49	3.1832 (15)	132

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