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Powder study of *N*-[2-(4-hydroxy-2-oxo-2,3-dihydro-1,3-benzothiazol-7-yl)ethyl]-3-[2-(2-naphthalen-1-ylethoxy)ethylsulfonyl]propylaminium benzoate

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Key indicators

Powder X-ray study
T = 295 K
Mean σ(C–C) = 0.089 Å
Disorder in solvent or counterion
R factor = 0.037
wR factor = 0.038
Data-to-parameter ratio = 6.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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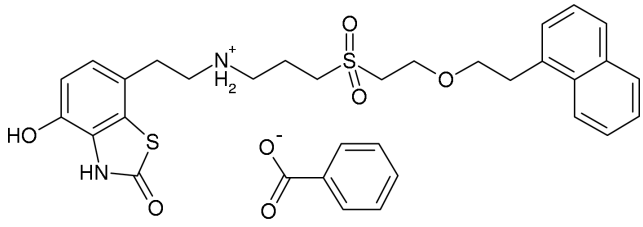
The crystal structure of the title compound, $C_{26}H_{31}N_2O_5S_2^+ \cdot C_7O_2H_5^-$, also known as AR-C69457CC, was solved by simulated annealing from laboratory X-ray powder diffraction data collected at room temperature to 2.1 Å resolution. Subsequent Rietveld refinement yielded an R_{wp} of 0.038 and site-occupancy factors for the disordered anion components of 0.5.

Comment

The title compound, (I), was synthesized by AstraZeneca during the development of a potential treatment for chronic obstructive pulmonary disease. The crystal structure of (I) was solved as part of a wider investigation into the application of simulated annealing to the problem of solving pharmaceutical crystal structures from laboratory X-ray powder diffraction data (Docherty, 2004). The hydrogen bonding and ring interactions in (I) are summarized in Fig. 3. Hydrogen bond '*a*' [O1···N2 = 2.82 (6) Å] links two cations to form a centrosymmetric dimer, within which the heterocyclic rings make face-to-face contact (*R*1···*R*1′ in Fig. 3) and the carbonyl O atom makes a close approach to the centroid of benzene ring *R*2′ [O1···centroid = 3.54 (3) Å and C1—O1···centroid = 95 (3)°]. The heterocyclic ring also engages in face-to-face contact with the C2–C7 benzene ring (Fig. 3, top right, *R*1···*R*2*a* and *R*2···*R*1*a*). The donor–acceptor distances for the three cation–anion hydrogen bonds '*b*' to '*d*' fall in the range 2.38 (12)–2.51 (13) Å and the hydrogen-bonding scheme is preserved on switching between the two half-occupancy anion sites. The naphthalene rings engage with each other in offset face-to-face interactions (Fig. 3, bottom right) and pack, along with the benzoate phenyl ring, to form a hydrophobic layer in the *ab* plane.



(I)

Experimental

A polycrystalline sample of the title compound was recrystallized from acetonitrile solution by slow evaporation at room temperature. Data were collected from a sample in a rotating 0.7 mm borosilicate glass capillary using a variable count time scheme (Hill & Madsen, 2002).

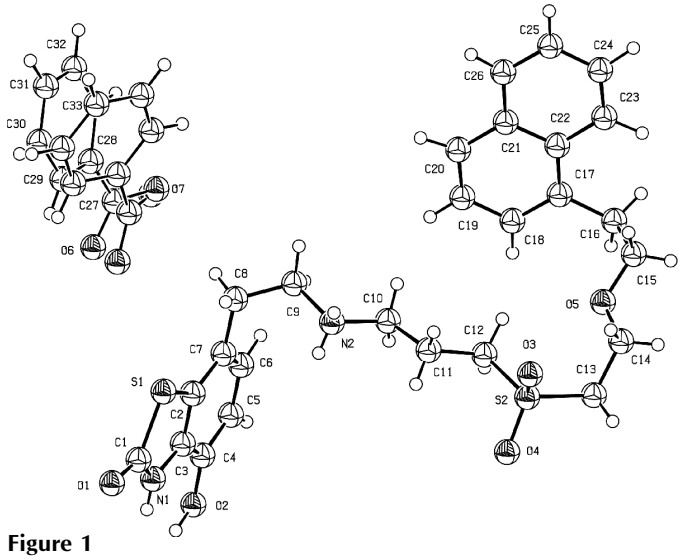


Figure 1
The atomic arrangement in (I), showing the anion disordered over two half-occupancy sites. Isotropic displacement spheres are shown at the 50% probability level.

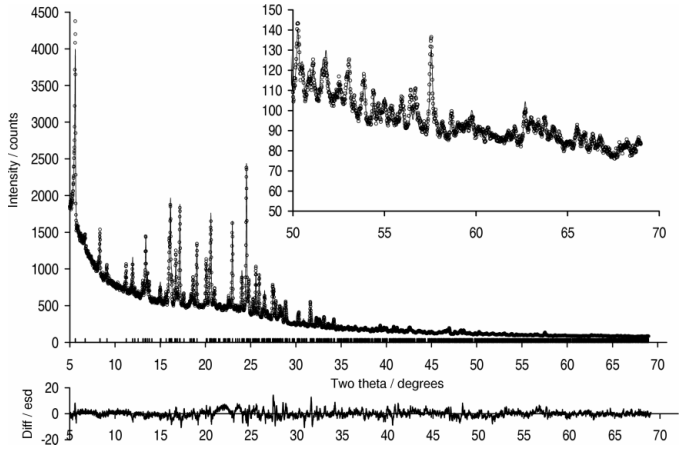


Figure 2
Final observed (points), calculated (line) and difference [$(y_{obs} - y_{calc})$/s.u.] profiles for the Rietveld refinement of (I). The reflection positions are shown by vertical bars.

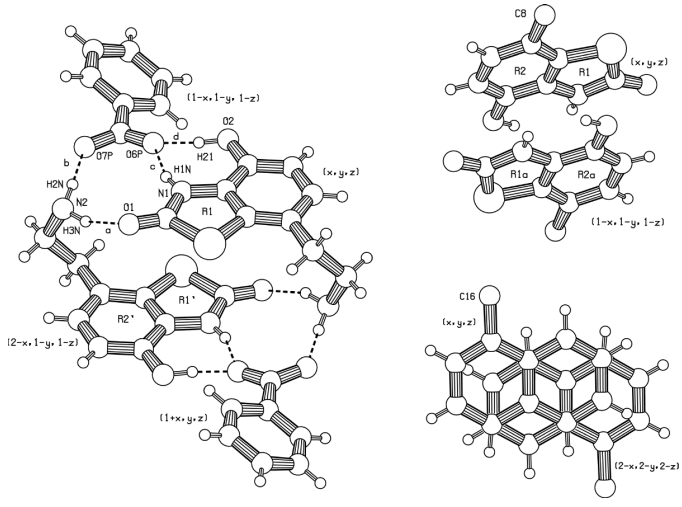


Figure 3
The hydrogen-bonding and ring interactions in (I), calculated and illustrated using *PLATON* (Spek, 2003; program version 280604).

Crystal data

$C_{26}H_{31}N_2O_5S_2^+\cdot C_7H_5O_2^-$	Cu $K\alpha_1$ radiation
M_r = 636.77	Cell parameters from 1347 reflections
Triclinic, $P\overline{1}$	θ = 2.5–34.5°
a = 7.63122 (17) Å	μ = 1.94 mm^{-1}
b = 13.66728 (32) Å	T = 295 K
c = 15.8058 (5) Å	White
α = 84.3849 (21)°	Specimen shape: cylinder
β = 87.4653 (19)°	12 × 0.7 × 0.7 mm
γ = 75.7135 (13)°	Specimen prepared at 295 K
V = 1589.52 (7) Å^3	Particle morphology: needle
Z = 2	
D_x = 1.328 Mg m^{-3}	

Data collection

Bruker AXS *D*8 Advance diffractometer	1347 measured reflections
Specimen mounting: 0.7 mm borosilicate capillary	$h = 0 \rightarrow 5$
Specimen mounted in transmission mode	$k = -9 \rightarrow 10$
	$l = -11 \rightarrow 11$
	$2\theta_{min}$ = 5, $2\theta_{max}$ = 69.°
	Increment in 2θ = 0.014°

Refinement

R_p = 0.037	Only coordinates of H atoms refined
R_{wp} = 0.038	$(\Delta/\sigma)_{max}$ = 0.049
R_{exp} = 0.015	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
S = 1.60	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
213 parameters	

The diffraction pattern indexed to a triclinic cell [$F(20)$ = 124.5, $M(20)$ = 33.5; *DICVOL*91 (Boultif & Louer, 1991)] and space group $P\overline{1}$ was assigned from volume considerations and a lack of systematic absences. The data set was background subtracted and truncated to 42° 2θ for Pawley fitting (Pawley, 1981; χ^2_{Pawley} = 2.7) and the structure solved using the simulated annealing (SA) global optimization procedure, described previously (David *et al.*, 1998), that is now implemented in the *DASH* computer program (David *et al.*, 2001). The SA structure solution involved the optimization of two fragments (the cation with 13 torsion angles plus the anion) totaling 26 degrees of freedom. The best SA solution had a favourable $\chi^2_{SA}/\chi^2_{Pawley}$ ratio of 5.7 and a chemically sensible packing arrangement, but suffered from a significant misfit to the data, even at modest 2θ angles. Rerunning the SA with the cation fixed in its previously determined position and optimizing the positions and orientations of two 50% occupancy anions halved the $\chi^2_{SA}/\chi^2_{Pawley}$ ratio to 2.9 and significantly improved the fit at lower 2θ angles. The solved structure was then refined against the full data set (5–69° 2θ) using a restrained Rietveld method (Rietveld, 1969) as implemented in *TOPAS* (Coelho, 2003), with the R_{wp} falling from 0.064 to 0.038 during the refinement. All cation atomic positions (including H atoms) were refined, subject to a series of restraints on bond lengths, angles and, where appropriate, planarity. The distance and angle restraints were based on a geometric analysis of five cations in four crystal structures (Docherty, 2004) closely related to the title compound, namely (*a*) 2-(4-hydroxy-2-oxo-2,3-dihydro-1,3-benzothiazol-7-yl)ethylammonium chloride, (*b*) the monohydrate of (*a*), (*c*) *N*-[2-(4-hydroxy-2-oxo-2,3-dihydro-1,3-benzothiazol-7-yl)ethyl]-3-[2-(2-(4-methylphenyl)ethoxy)ethylsulfamoyl]propylaminium besilate besilate and (*d*) the tosilate analogue of (*c*). This was supplemented by a geometric analysis of naphthalene rings using the knowledge base, *MOGUL* (Bruno *et al.*, 2004). The half-occupancy anions could not be refined reliably using the strategy just described and were therefore refined as rigid bodies. A March–Dollase correction of intensities for preferred orientation (Dollase, 1986) was applied and

the refined value of the preferred orientation coefficient along the [001] direction was 1.13 (1).

Data collection: *DIFFRAC Plus XRD Commander* (Kienle & Jacob, 2003); cell refinement: *TOPAS* (Coelho, 2003); data reduction: *DASH* (David *et al.*, 2001); program(s) used to solve structure: *DASH*; program(s) used to refine structure: *TOPAS*; molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *enCIFer* (Cambridge Crystallographic Data Centre, 2004).

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supporting information

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Crystal data

$C_{26}H_{31}N_2O_5S_2^+ \cdot C_7H_5O_2^-$
$M_r = 636.77$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.63122\ (17)$ Å
$b = 13.6673\ (3)$ Å
$c = 15.8058\ (5)$ Å
$\alpha = 84.385\ (2)°$
$\beta = 87.4653\ (19)°$
$\gamma = 75.7135\ (13)°$
$V = 1589.52\ (7)$ Å^3
$Z = 2$
$F(000) = 672$
$D_x = 1.328$ Mg m^{-3}
Cu $K\alpha_1$ radiation, $\lambda = 1.54056$ Å
$\mu = 1.94$ mm^{-1}
$T = 295$ K
Particle morphology: needle
white
cylinder, 12 × 0.7 mm
Specimen preparation: Prepared at 295 K

Data collection

Bruker AXS D8 Advance diffractometer
Radiation source: sealed X-ray tube
Primary focussing, Ge 111 monochromator
Specimen mounting: 0.7 mm borosilicate capillary
Data collection mode: transmission
Scan method: step
$2\theta_{min} = 5°$, $2\theta_{max} = 69.000°$, $2\theta_{step} = 0.014°$

Refinement

Least-squares matrix: selected elements only
$R_p = 0.037$
$R_{wp} = 0.038$
$R_{exp} = 0.015$
4480 data points
Profile function: Fundamental parameters with axial divergence correction
213 parameters
194 restraints
1 constraint
Only H-atom coordinates refined
Weighting scheme based on measured s.u.'s
$(\Delta/\sigma)_{max} = 0.049$
Background function: Chebyshev polynomial
Preferred orientation correction: A March-Dollase correction of intensities for preferred orientation was applied. The refined value of the preferred orientation coefficient along the [0 0 1] direction was 1.13(1).

Special details

Geometry. Bond distances, bond angles, torsion angles and H-bond geometries were calculated using *PLATON* (Spek, 2003; program version 280604)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.8648 (18)	0.4241 (10)	0.5995 (11)	0.0588 (15)*	
S2	1.249 (2)	0.9911 (10)	0.5989 (12)	0.0588 (15)*	
O1	0.993 (3)	0.301 (2)	0.478 (2)	0.0588 (15)*	
O2	0.555 (4)	0.625 (2)	0.347 (3)	0.0588 (15)*	
O3	1.381 (3)	0.9424 (19)	0.661 (2)	0.0588 (15)*	
O4	1.277 (3)	0.956 (2)	0.515 (2)	0.0588 (15)*	
O5	1.062 (4)	1.182 (3)	0.711 (2)	0.0588 (15)*	
N1	0.792 (7)	0.453 (4)	0.441 (3)	0.0588 (15)*	
N2	0.863 (6)	0.734 (4)	0.687 (3)	0.0588 (15)*	
C1	0.896 (7)	0.382 (4)	0.495 (3)	0.0588 (15)*	
C2	0.713 (7)	0.537 (4)	0.563 (4)	0.0588 (15)*	
C3	0.691 (7)	0.539 (4)	0.476 (4)	0.0588 (15)*	
C4	0.576 (6)	0.622 (4)	0.432 (5)	0.0588 (15)*	
C5	0.483 (6)	0.701 (4)	0.478 (4)	0.0588 (15)*	
H5	0.41 (5)	0.76 (3)	0.45 (3)	0.0760*	
C6	0.510 (6)	0.698 (4)	0.565 (4)	0.0588 (15)*	
H6	0.45 (6)	0.75 (3)	0.60 (3)	0.0760*	
C7	0.627 (7)	0.618 (4)	0.609 (4)	0.0588 (15)*	
C8	0.656 (6)	0.616 (5)	0.704 (4)	0.0588 (15)*	
H8A	0.76 (5)	0.56 (3)	0.72 (3)	0.0760*	
H8B	0.55 (5)	0.61 (3)	0.73 (3)	0.0760*	
C9	0.693 (8)	0.713 (5)	0.727 (4)	0.0588 (15)*	
H9A	0.59 (5)	0.77 (3)	0.71 (3)	0.0760*	
H9B	0.70 (6)	0.71 (3)	0.79 (3)	0.0760*	
C10	0.856 (7)	0.845 (5)	0.678 (4)	0.0588 (15)*	
H10A	0.81 (5)	0.87 (3)	0.73 (3)	0.0760*	
H10B	0.78 (6)	0.88 (3)	0.63 (3)	0.0760*	
C11	1.040 (7)	0.864 (4)	0.656 (4)	0.0588 (15)*	
H11A	1.09 (5)	0.83 (3)	0.61 (3)	0.0760*	
H11B	1.12 (5)	0.84 (3)	0.70 (3)	0.0760*	
C12	1.036 (7)	0.975 (5)	0.638 (4)	0.0588 (15)*	
H12A	1.01 (6)	1.01 (3)	0.69 (3)	0.0760*	
H12B	0.95 (5)	1.01 (3)	0.60 (3)	0.0760*	
C13	1.241 (6)	1.123 (4)	0.589 (4)	0.0588 (15)*	
H13A	1.34 (6)	1.13 (3)	0.56 (3)	0.0760*	
H13B	1.13 (5)	1.16 (3)	0.56 (3)	0.0760*	
C14	1.241 (7)	1.162 (4)	0.675 (5)	0.0588 (15)*	
H14A	1.32 (5)	1.11 (3)	0.71 (3)	0.0760*	
H14B	1.28 (5)	1.22 (3)	0.67 (3)	0.0760*	
C15	1.033 (7)	1.240 (4)	0.781 (5)	0.0588 (15)*	
H15A	1.07 (5)	1.30 (3)	0.77 (3)	0.0760*	
H15B	1.10 (5)	1.20 (3)	0.83 (3)	0.0760*	
C16	0.832 (8)	1.267 (4)	0.803 (3)	0.0588 (15)*	
H16A	0.81 (6)	1.32 (3)	0.84 (3)	0.0760*	
H16B	0.77 (5)	1.29 (3)	0.75 (2)	0.0760*	

C17	0.773 (6)	1.176 (4)	0.846 (4)	0.0588 (15)*	
C18	0.705 (7)	1.116 (5)	0.797 (3)	0.0588 (15)*	
H18	0.69 (5)	1.13 (3)	0.74 (3)	0.0760*	
C19	0.650 (7)	1.033 (4)	0.835 (4)	0.0588 (15)*	
H19	0.61 (5)	0.99 (3)	0.80 (3)	0.0760*	
C20	0.673 (7)	1.004 (3)	0.920 (5)	0.0588 (15)*	
H20	0.64 (5)	0.94 (3)	0.94 (4)	0.0760*	
C21	0.747 (6)	1.062 (4)	0.972 (5)	0.0588 (15)*	
C22	0.802 (6)	1.148 (4)	0.934 (4)	0.0588 (15)*	
C23	0.876 (6)	1.205 (4)	0.987 (6)	0.0588 (15)*	
H23	0.91 (6)	1.27 (3)	0.96 (3)	0.0760*	
C24	0.902 (7)	1.175 (5)	1.072 (5)	0.0588 (15)*	
H24	0.94 (6)	1.22 (3)	1.11 (3)	0.0760*	
C25	0.836 (7)	1.096 (5)	1.109 (3)	0.0588 (15)*	
H25	0.85 (5)	1.08 (3)	1.17 (3)	0.0760*	
C26	0.772 (7)	1.035 (4)	1.061 (5)	0.0588 (15)*	
H26	0.74 (5)	0.98 (3)	1.09 (3)	0.0760*	
H3N	0.88 (6)	0.71 (3)	0.64 (3)	0.0760*	
H2N	0.96 (5)	0.70 (3)	0.72 (3)	0.0760*	
H1N	0.79 (7)	0.45 (4)	0.39 (3)	0.0760*	
H21	0.63 (6)	0.57 (3)	0.32 (3)	0.0760*	
O6	0.172 (13)	0.504 (9)	0.726 (8)	0.0588 (15)*	0.5
O7	0.117 (13)	0.632 (9)	0.803 (8)	0.0588 (15)*	0.5
C27	0.161 (13)	0.538 (9)	0.798 (8)	0.0588 (15)*	0.5
C28	0.199 (13)	0.467 (9)	0.876 (8)	0.0588 (15)*	0.5
C29	0.318 (13)	0.373 (9)	0.873 (8)	0.0588 (15)*	0.5
C30	0.353 (13)	0.306 (9)	0.946 (8)	0.0588 (15)*	0.5
C31	0.270 (13)	0.335 (9)	1.023 (8)	0.0588 (15)*	0.5
C32	0.150 (13)	0.429 (9)	1.026 (8)	0.0588 (15)*	0.5
C33	0.114 (13)	0.495 (9)	0.953 (8)	0.0588 (15)*	0.5
H29	0.376 (13)	0.353 (9)	0.820 (8)	0.0760*	0.5
H30	0.435 (13)	0.241 (9)	0.944 (8)	0.0760*	0.5
H31	0.294 (13)	0.289 (9)	1.073 (8)	0.0760*	0.5
H32	0.094 (13)	0.448 (9)	1.079 (8)	0.0760*	0.5
H33	0.032 (13)	0.559 (9)	0.955 (8)	0.0760*	0.5
O6P	0.268 (13)	0.520 (9)	0.715 (8)	0.0588 (15)*	0.5
O7P	0.093 (13)	0.632 (9)	0.790 (8)	0.0588 (15)*	0.5
C27P	0.213 (13)	0.550 (9)	0.786 (8)	0.0588 (15)*	0.5
C28P	0.289 (13)	0.490 (9)	0.866 (8)	0.0588 (15)*	0.5
C29P	0.346 (13)	0.385 (9)	0.869 (8)	0.0588 (15)*	0.5
C30P	0.415 (13)	0.329 (9)	0.943 (8)	0.0588 (15)*	0.5
C31P	0.430 (13)	0.379 (9)	1.014 (8)	0.0588 (15)*	0.5
C32P	0.375 (13)	0.484 (9)	1.011 (8)	0.0588 (15)*	0.5
C33P	0.303 (13)	0.539 (9)	0.937 (8)	0.0588 (15)*	0.5
H29P	0.336 (13)	0.352 (9)	0.820 (8)	0.0760*	0.5
H30P	0.453 (13)	0.258 (9)	0.945 (8)	0.0760*	0.5
H31P	0.478 (13)	0.340 (9)	1.065 (8)	0.0760*	0.5
H32P	0.385 (13)	0.517 (9)	1.060 (8)	0.0760*	0.5

H33P	0.265 (13)	0.611 (9)	0.935 (8)	0.0760*	0.5

Geometric parameters (Å, º)

S1—C1	1.79 (5)	C10—H10B	1.0 (4)
S1—C2	1.75 (6)	C11—H11A	0.9 (4)
S2—O3	1.43 (3)	C11—H11B	0.9 (5)
S2—O4	1.44 (4)	C12—H12A	1.0 (5)
S2—C12	1.77 (6)	C12—H12B	0.9 (4)
S2—C13	1.78 (5)	C13—H13B	1.0 (4)
O1—C1	1.22 (6)	C13—H13A	0.9 (5)
O2—C4	1.36 (9)	C14—H14B	0.9 (5)
O5—C14	1.43 (7)	C14—H14A	1.0 (5)
O5—C15	1.40 (8)	C15—H15A	0.9 (4)
N1—C1	1.35 (7)	C15—H15B	1.0 (5)
N1—C3	1.39 (8)	C16—H16A	1.0 (5)
N2—C10	1.50 (8)	C16—H16B	1.0 (3)
N2—C9	1.50 (8)	C18—H18	0.9 (5)
C2—C3	1.39 (9)	C19—H19	1.0 (3)
C2—C7	1.39 (8)	C20—H20	1.0 (5)
C3—C4	1.40 (8)	C23—H23	1.0 (5)
C4—C5	1.39 (8)	C24—H24	1.0 (5)
C5—C6	1.40 (9)	C25—H25	1.0 (5)
C6—C7	1.38 (8)	C26—H26	0.9 (4)
C7—C8	1.52 (9)	O6—C27	1.26
C8—C9	1.50 (9)	O6P—C27P	1.25
C10—C11	1.51 (8)	O7—C27	1.25
C11—C12	1.51 (9)	O7P—C27P	1.27
C13—C14	1.51 (10)	C27—C28	1.48
C15—C16	1.52 (8)	C27P—C28P	1.50
C16—C17	1.52 (8)	C28—C29	1.38
C17—C18	1.38 (8)	C28P—C29P	1.39
C17—C22	1.42 (9)	C28—C33	1.40
C18—C19	1.38 (8)	C28P—C33P	1.38
C19—C20	1.37 (10)	C29—C30	1.39
C20—C21	1.42 (9)	C29P—C30P	1.38
C21—C26	1.43 (11)	C30—C31	1.39
C21—C22	1.42 (8)	C30P—C31P	1.39
C22—C23	1.42 (9)	C31—C32	1.39
C23—C24	1.37 (12)	C31P—C32P	1.39
C24—C25	1.37 (9)	C32—C33	1.39
C25—C26	1.37 (9)	C32P—C33P	1.38
O2—H21	1.0 (4)	C29—H29	0.96
N1—H1N	0.8 (5)	C29P—H29P	0.95
N2—H2N	0.9 (5)	C30—H30	0.95
N2—H3N	0.8 (4)	C30P—H30P	0.94
C5—H5	0.9 (4)	C31—H31	0.96
C6—H6	1.0 (5)	C31P—H31P	0.96

C8—H8A	1.0 (4)	C32—H32	0.95
C8—H8B	0.9 (4)	C32P—H32P	0.95
C9—H9A	1.0 (4)	C33—H33	0.94
C9—H9B	1.0 (5)	C33P—H33P	0.95
C10—H10A	0.9 (5)		

C1—S1—C2	92 (3)	C17—C22—C21	119 (5)
O3—S2—O4	117 (2)	C22—C23—C24	121 (6)
O3—S2—C12	108 (3)	C23—C24—C25	120 (6)
O3—S2—C13	108 (2)	C24—C25—C26	121 (6)
O4—S2—C12	108 (2)	C21—C26—C25	120 (5)
O4—S2—C13	108 (3)	O6—C27—O7	119
C12—S2—C13	108 (3)	O6P—C27P—O7P	120
C14—O5—C15	116 (4)	O6—C27—C28	120
C1—N1—C3	116 (5)	O6P—C27P—C28P	120
C9—N2—C10	112 (5)	O7—C27—C28	120
O1—C1—N1	127 (5)	O7P—C27P—C28P	120
S1—C1—N1	109 (4)	C27—C28—C29	120
S1—C1—O1	124 (4)	C27P—C28P—C29P	120
S1—C2—C3	110 (4)	C27—C28—C33	120
C3—C2—C7	122 (5)	C27P—C28P—C33P	120
S1—C2—C7	128 (5)	C29—C28—C33	120
N1—C3—C2	114 (5)	C29P—C28P—C33P	120
C2—C3—C4	121 (5)	C28—C29—C30	121
N1—C3—C4	126 (6)	C28P—C29P—C30P	120
O2—C4—C5	121 (5)	C29—C30—C31	120
O2—C4—C3	121 (5)	C29P—C30P—C31P	119
C3—C4—C5	118 (7)	C30—C31—C32	120
C4—C5—C6	120 (5)	C30P—C31P—C32P	121
C5—C6—C7	122 (5)	C31—C32—C33	120
C2—C7—C8	121 (5)	C31P—C32P—C33P	120
C6—C7—C8	122 (5)	C28—C33—C32	120
C2—C7—C6	117 (6)	C28P—C33P—C32P	120
C7—C8—C9	112 (5)	C28—C29—H29	120
N2—C9—C8	114 (5)	C28P—C29P—H29P	119
N2—C10—C11	112 (5)	C30—C29—H29	119
C10—C11—C12	113 (5)	C30P—C29P—H29P	120
S2—C12—C11	111 (4)	C29—C30—H30	120
S2—C13—C14	111 (4)	C29P—C30P—H30P	120
O5—C14—C13	110 (4)	C31—C30—H30	120
O5—C15—C16	109 (5)	C31P—C30P—H30P	120
C15—C16—C17	111 (4)	C30—C31—H31	120
C18—C17—C22	121 (5)	C30P—C31P—H31P	119
C16—C17—C18	119 (5)	C32—C31—H31	121
C16—C17—C22	120 (5)	C32P—C31P—H31P	120
C17—C18—C19	120 (5)	C31—C32—H32	119
C18—C19—C20	121 (5)	C31P—C32P—H32P	120
C19—C20—C21	120 (5)	C33—C32—H32	121

C20—C21—C26	122 (5)	C33P—C32P—H32P	121
C22—C21—C26	119 (5)	C28—C33—H33	120
C20—C21—C22	119 (6)	C28P—C33P—H33P	120
C17—C22—C23	123 (5)	C32—C33—H33	120
C21—C22—C23	118 (6)	C32P—C33P—H33P	120

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H3*N*···O1^{i}	0.8 (4)	2.1 (5)	2.82 (6)	151 (12)
N2—H2*N*···O7*P*^{ii}	0.9 (5)	1.6 (4)	2.51 (13)	167 (14)
N1—H1*N*···O6*P*^{iii}	0.8 (5)	1.7 (5)	2.50 (13)	161 (13)
O2—H21···O6*P*^{iii}	1.0 (4)	1.4 (5)	2.38 (12)	174 (13)
N2—H2*N*···O7^{ii}	0.9 (5)	1.8 (4)	2.75 (13)	168 (14)
N1—H1*N*···O6^{iii}	0.8 (5)	1.9 (5)	2.67 (13)	152 (13)
O2—H21···O6^{iii}	1.0 (4)	1.8 (5)	2.67 (12)	158 (12)

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