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Crystal structures of salts of bedaquiline

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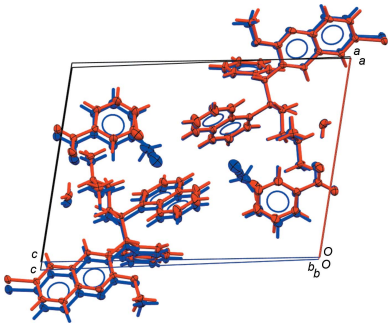
Supporting information: this article has supporting information at journals.iucr.org/c

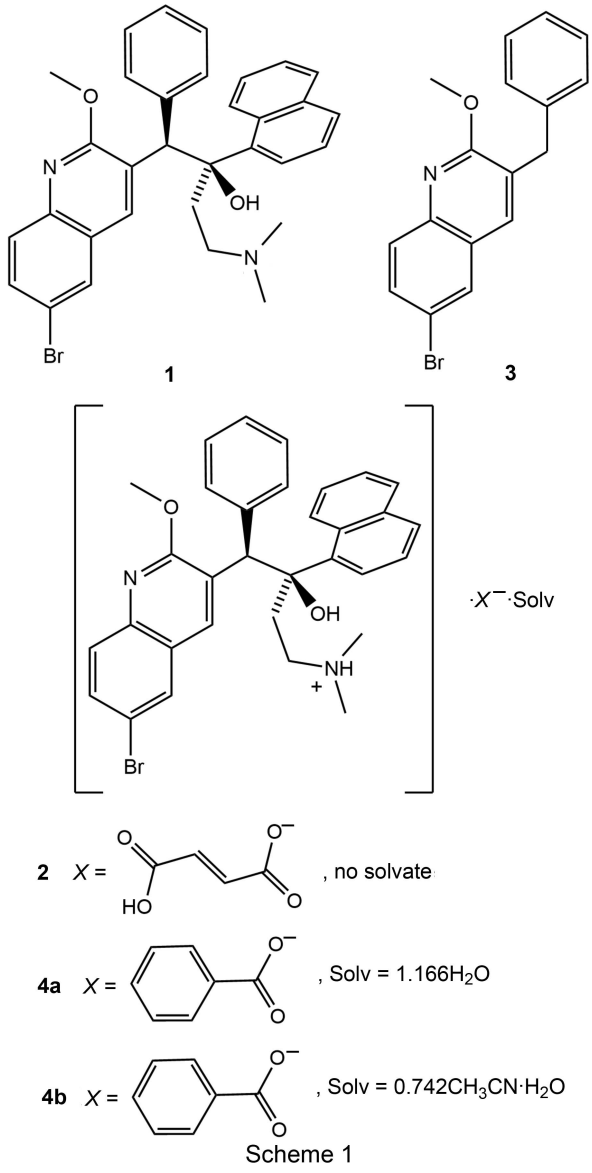
Bedaquiline [systematic name: 1-(6-bromo-2-methoxyquinolin-3-yl)-4-(dimethylamino)-2-(naphthalen-1-yl)-1-phenylbutan-2-ol, $C_{32}H_{31}BrN_2O_2$] is one of two important new drugs for the treatment of drug-resistant tuberculosis (TB). It is marketed in the US as its fumarate salt {systematic name: [4-(6-bromo-2-methoxyquinolin-3-yl)-3-hydroxy-3-(naphthalen-1-yl)-4-phenylbutyl]-dimethylazanium 3-carboxyprop-2-enoate, $C_{32}H_{32}BrN_2O_2^+\cdot C_4H_3O_4^-$}, and about a dozen other salts of bedaquiline have been described in patent literature, but none have so far been structurally described. In a first communication, we present the crystal structure of bedaquilinium fumarate and of two new benzoate salts, as well as that of a degradation product of the reaction of bedaquilinium fumarate with sodium ethoxide, 3-benzyl-6-bromo-2-methoxyquinoline, $C_{17}H_{14}BrNO$. The fumarate and benzoate salts both feature cations monoprotonated at the dimethylamino group. The much less basic quinoline N atom remains unprotonated. Both salts feature a 1:1 cation-to-anion ratio, with the fumarate being present as monoanionic hydrofumarate. The conformations of the cations are compared to that of free base bedaquiline and with each other. The flexible backbone of the bedaquiline structure leads to a landscape of conformations with little commonalities between the bedaquiline entities in the various structures. The conformations are distinctively different for the two independent molecules of the free base, the two independent molecules of the hydrofumarate salt, and the one unique cation of the benzoate salt. Packing of the salts is dominated by hydrogen bonding. Hydrogen-bonding motifs, as well as the larger hydrogen-bonded entities within the salts, are quite similar for the salts, despite the vastly differing conformations of the cations, and both the hydrofumarate and the benzoate structure feature chains of hydrogen-bonded anions that are surrounded by and hydrogen bonded to the larger bedaquilinium cations, leading to infinite broad ribbons of anions, cations, and (for the benzoate salt) water molecules. The benzoate salt was isolated in two forms: as a 1.17-hydrate ($C_{32}H_{32}BrN_2O_2^+\cdot C_7H_5O_2^-\cdot 1.166H_2O$), obtained from acetone or propanol solution, with one fully occupied water molecule tightly integrated into the hydrogen-bonding network of anions and cations, and one partially occupied water molecule [refined occupancy 16.6 (7)%], only loosely hydrogen bonded to the quinoline N atom. The second form is an acetonitrile solvate ($C_{32}H_{32}BrN_2O_2^+\cdot C_7H_5O_2^-\cdot 0.742CH_3CN\cdot H_2O$), in which the partially occupied water molecule is replaced by a 74.2 (7)%-occupied acetonitrile molecule. The partial occupancy induces disorder for the benzoate phenyl ring. The acetonitrile solvate is unstable in atmosphere and converts into a form not distinguishable by powder XRD from the 1.17-hydrate.



1. Introduction

Bedaquiline, **1**, is one of two important new drugs for the treatment of drug-resistant tuberculosis (TB). Bedaquiline is

marketed in the US as the fumarate salt (**2**) with the trade name Sirturo (Brigden *et al.*, 2015). The fumarate salt is described in US Patent 8 546 428 (Hegyi *et al.*, 2013). The citrate, sulfate, phosphate, and tartrate salts are described in two other patents (Zvatora, Dammer, Krejcik *et al.*, 2016; Zvatora, Dammer, Ridvan *et al.*, 2016). However, none of these salts has been structurally described in detail. For the fumarate, as well as one each of the two sulfate and citrate salts, well-resolved powder X-ray patterns have been reported, but the structures were not solved and no single-crystal data are reported. For the remaining salts (the phosphate and tartrate salts, and the second sulfate and citrate polymorphs), the powder patterns indicate the samples to have either extremely small particle distributions or to be entirely amorphous. Detailed structural data are reported solely for the free base form of bedaquiline (Petit *et al.*, 2007).



Scheme 1

Bedaquiline features two basic N atoms that are amenable to protonation, *i.e.* the tertiary amine appended to the dangling ethylene group and the pyridine N atom. The two sites have distinctly different basicities and selective protonation of only the more basic amine site should be possible. Salts of both mono- and dicationic bedaquilinium ions can thus be formed, depending on the strength and amount of acid used for salt formation. The formation of cocrystals (with no or incomplete proton transfer) can also be imagined.

The NMR data reported in the patent publications indicate bedaquilinium fumarate to have a 1:1 anion-to-cation ratio. Whether the bedaquiline is protonated once or twice (and the fumarate deprotonated once or twice) had not been disclosed. For the sulfate salts, a 1:1 molar ratio of bedaquiline-to-sulfuric acid was used, but the anion-to-cation ratio in the salt was not determined. The given reaction yields, assuming a 1:1 salt, are around 33%, which would allow formation of a 1:2, a 1:1, or a 2:1 salt. The patent specifically states a wide range for the molar ratio of bedaquiline to sulfuric acid: 'The molar ratio of bedaquiline:sulfuric acid may be in the range of 10:1 to 1:3, preferably 1:1, 2:1, and 1:2'. Similar statements have been made for the tartrate salts, one of the citrate salts, and the phosphate salt. No elemental analysis data are given to support any of the possible ratios, thus leaving the stoichiometry and the overall nature of the presented salts in question. The possibility of hydrate or solvate formation was also not properly addressed in the patent claims.

This lack of structural knowledge and even of basic chemical composition frustrates the understanding of the chemical, physical, and physiological properties of bedaquiline and its derivatives. To reduce this paucity of information on the bedaquiline system, there is interest in developing additional salts of bedaquiline and obtaining detailed analysis and structural data for these compounds, to better understand and possibly improve their properties, such as solubility, which in turn affect pharmacokinetics and dosage. Additional reasons for this study include finding a bedaquiline salt with improved stability and hygroscopicity.

2. Experimental

Melting points were determined using a Thomas Hoover Capillary Melting Point apparatus and are uncorrected. NMR data were collected in acetonitrile-d_3 (ACN-d_3) using a Bruker DRX-500 spectrometer and were referenced against the residual nondeuterated solvent peak.

Benzoic acid was purchased from Mallinckrodt, acetone from Fischer Chemicals, and acetonitrile from VWR Chemicals. Hydrochloride in methanol 1.25 *M* was obtained from Fluka. Bedaquiline fumarate was obtained from Johnson & Johnson. All chemicals were used as received without further purification.

Polarized light microscopy images were collected using an Olympus Series BX51TRF (Olympus America Inc., Melville, NY) polarized light microscope equipped with 12 V/100 W illumination; an Achromat 0.9 NA polarized-light condenser; Olympus Series UPlanFL N objectives: 40X/0.75 NA, 20X/0.50 NA, 10X/0.30 NA, and 4X/0.13 NA; an intermediate tube with variable position analyzer and compensator; and a trinocular viewing head with a Lumenera Series Infinity X

(Teledyne Lumenera, Ottawa, Ontario, Canada) digital camera using *Infinity* (Version 6.5.6) and *Infinity Analyze* software (Version 7.0.2.930, Build date 01-Feb-2020). A small portion of sample was placed on a cleaned microscope slide and a No. 1 1/2-cover glass placed over the sample. Mineral oil, USP (CAS: 8042-47-5), was allowed to cover the sample by capillarity. Images were acquired as a collection of three: (i) plane polarized light, (ii) crossed polarized light, and (iii) crossed polarized light with a first-order red compensator. Microscopy observations revealed crystal habits for bedaquilinium benzoate powders as birefringent platy anhedral agglomerates that are softly bound and easily dispersed under light pressure from a tungsten needle on the cover glass. A representative collection of images is given in the supporting information.

IR microspectroscopy experiments were conducted using a Smiths Detection (Danbury, CT) IlluminatIR 1.5 IR Microspectrometer accessory on an Olympus Series BX41TF polarized-light microscope (Olympus America Inc., Melville, NY), which provided the base optical platform. The IlluminatIR 1.5 is equipped with a gray-body ceramic IR source, a 60° Michelson Interferometer with a zinc–selenide (ZnSe) beam splitter, a 4 wavenumber (cm^{-1}) spectral resolution, and a 0.25 × 0.25 mm liquid-nitrogen-cooled mercury cadmium telluride (MCT) photoconductive detector, and the sample area was defined using a fixed circular 100 µm aperture. The IlluminatIR 1.5 is computer-interfaced using universal serial bus (USB) communications with Smiths Detection *QualID* App (Version 2.51, 2005) software. Advanced data processing was conducted using either Thermo Galactic spectral analysis software packages *GRAMS/AI* and *SpectralID*, or Thermo Fisher Scientific *OMNIC* software (Version 9.11.706, 2020). IR analyses were performed by reflection/absorption (R/A) using an all-reflecting objective (ARO, 15X, 0.88 NA). A small amount of sample was transferred to a low-E microscope slide (Smiths Detection P/N: 006-4013) and dispersed to a thin layer. IR microprobe analyses were conducted on what appeared microscopically to be a single crystal. An FT–IR spectral background was collected immediately prior to each sample spectral analysis.

Powder X-ray diffraction (XRD) data were collected in focusing mode on a PANalytical Empyrean X-ray diffractometer equipped with Bragg–Brentano HD optics, a sealed-tube copper X-ray source (λ = 1.54178 Å), Soller slits on both the incident and receiving optics sides, and a PixCel3D Medipix detector. Samples were hand ground using an agate mortar and pestle, and packed into a silicon single-crystal zero-background sample holder, 16 mm wide and 0.25 mm deep. Antiscatter slits and divergence slits, as well as the mask, were chosen based on sample area and starting θ angle. Data were collected between 4 and 40° in 2θ under ambient conditions using the PANalytical *Data Collector* software (PANalytical, 2015). Rietveld refinements were performed against the 150 K models of the single-crystal structure data sets using *HighScore* (PANalytical, 2015) software. Refinement of preferred orientation was included using a spherical harmonics model. Plots of Rietveld fits for all compounds are given in the supporting information.

2.1. Synthesis and crystallization

2.1.1. Free base bedaquiline (1). The free base used during synthesis was prepared by extracting a CH_2Cl_2 solution of the fumarate three times with saturated $NaHCO_3$ solution (Rombouts *et al.*, 2016). The identity and purity of the free base thus afforded from the material supplied by Johnson & Johnson was verified using NMR spectroscopy [m.p. 175–176 °C; literature value 181°C (Zvatora, Dammer, Ridvan *et al.*, 2016)]. ^{1}H NMR (500 MHz, ACN-d_3): δ 8.82 (*s*, 1H), 8.66 (*d*, *J* = 8.7 Hz, 1H), 8.03 (*s*, 1H), 8.02 (*d*, *J* = 7.3 Hz, 1H), 7.87 (*d*, *J* = 8.0 Hz, 1H), 7.66 (*t*, *J* = 7.7 Hz, 4H), 7.49 (*t*, *J* = 7.7 Hz, 1H), 7.30 (*m*, 3H), 6.87 (*m*, 3H), 5.88 (*s*, 1H), 4.20 (*s*, 3H), 2.52 (*d*, *J* = 14.6 Hz, 1H), 2.01 (*m*, 2H), 1.89 (*m*, 7H).

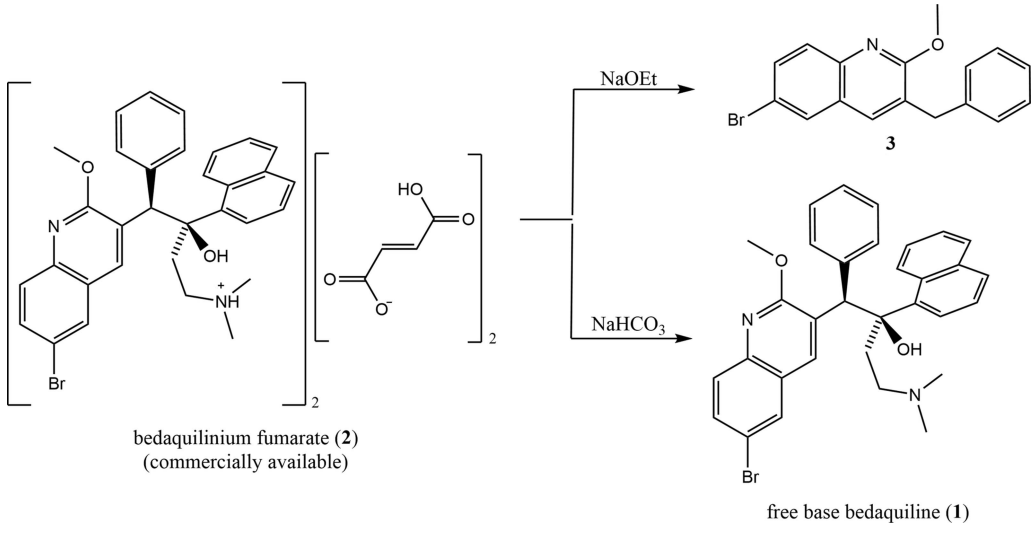


Figure 1
Isolation of free base bedaquiline (**1**) from commercially available bedaquilinium fumarate (**2**), and decomposition to **3**.

Table 1
Experimental details.

Experiments were carried out at 150 K. Absorption was corrected for by multi-scan methods (*SADABS2016*; Krause *et al.*, 2015).

	1	**2**	**3**
Crystal data			
Chemical formula	$C_{32}H_{31}BrN_2O_2$	$C_{32}H_{32}BrN_2O_2^+\cdot C_4H_3O_4^-$	$C_{17}H_{14}BrNO$
M_r	555.50	671.57	328.20
Crystal system, space group	Orthorhombic, $P2_12_12_1$	Monoclinic, $P2_1$	Orthorhombic, $P2_12_12_1$
a, b, c (Å)	11.1584 (8), 13.6425 (14), 36.061 (4)	16.4556 (6), 10.3205 (3), 20.1636 (8)	4.3606 (6), 10.820 (2), 29.886 (11)
α, β, γ (°)	90, 90, 90	90, 109.1832 (15), 90	90, 90, 90
V (Å^3)	5489.5 (9)	3234.2 (2)	1410.1 (6)
Z	8	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	1.53	1.32	2.91
Crystal size (mm)	0.21 × 0.13 × 0.05	0.45 × 0.37 × 0.17	0.41 × 0.06 × 0.05
Data collection			
Diffractometer	Bruker D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD)	Bruker D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD)	Bruker D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD)
T_{min}, T_{max}	0.603, 0.747	0.438, 0.495	0.658, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	66520, 17893, 12296	115858, 24622, 18572	28030, 5125, 4504
R_{int}	0.052	0.040	0.037
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.770	0.771	0.768
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.111, 1.03	0.043, 0.117, 1.06	0.023, 0.057, 1.05
No. of reflections	17893	24622	5125
No. of parameters	675	837	196
No. of restraints	0	1	0
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement	Only H-atom displacement parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.48, −0.58	1.23, −1.28	0.28, −0.38
Absolute structure	Flack x determined using 4397 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)	Flack x determined using 7327 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)	Flack x determined using 1685 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.034 (3)	−0.0144 (14)	−0.011 (3)

	4a	**4b**
Crystal data		
Chemical formula	$C_{32}H_{32}BrN_2O_2^+\cdot C_7H_5O_2^-\cdot 1.166H_2O$	$C_{32}H_{32}BrN_2O_2^+\cdot C_7H_5O_2^-\cdot 0.742C_2H_3N\cdot H_2O$
M_r	698.70	726.10
Crystal system, space group	Monoclinic, $P2_1$	Monoclinic, $P2_1$
a, b, c (Å)	12.6384 (5), 7.9259 (3), 17.5249 (8)	12.8661 (8), 8.0386 (5), 17.4704 (10)
α, β, γ (°)	90, 99.8450 (17), 90	90, 101.093 (3), 90
V (Å^3)	1729.63 (12)	1773.13 (19)
Z	2	2
Radiation type	Mo $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	1.24	1.97
Crystal size (mm)	0.55 × 0.21 × 0.13	0.31 × 0.05 × 0.05
Data collection		
Diffractometer	Bruker D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD)	Bruker D8 Quest diffractometer with PhotonIII_C14 charge-integrating and photon counting pixel array detector
T_{min}, T_{max}	0.638, 0.746	0.599, 0.754
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	80228, 13080, 10456	39739, 7360, 6750
R_{int}	0.049	0.060
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.770	0.639
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.073, 1.03	0.035, 0.085, 1.06
No. of reflections	13080	7360
No. of parameters	445	515
No. of restraints	5	195
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement

Table 1 (continued)

	4a	4b
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.48	0.40, −0.50
Absolute structure	Flack x determined using 4051 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)	Flack x determined using 2778 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.006 (3)	0.004 (8)

Computer programs: *APEX3* (Bruker, 2019), *SAINT* (Bruker, 2019), *SHELXS97* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015), *shelXle* (Hübschle *et al.*, 2011), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

Single crystals were grown by dissolving bedaquiline (30 mg, 0.054 mmol) in acetone (1 ml) in a 5 ml scintillation vial and the solution was allowed to evaporate slowly to obtain medium-sized plate-shaped crystals of **1**.

2.1.2. Decomposition of bedaquiline fumarate by sodium ethoxide. Sodium ethoxide (1.5 g, 22.0 mmol) was dissolved in EtOH (20 ml). The resulting solution was added to a solution of bedaquiline fumarate (5 g, 7.44 mmol) in ACN/EtOH (50 ml, 1:1 *v*/*v*). After 1 h, water was added slowly and the resulting mixture extracted with EtOAc. The combined organic layers were dried ($MgSO_4$) and then concentrated to provide a colorless crystalline material that was found by IR and NMR spectroscopies to not match free base bedaquiline. Individual crystals were identified as 3-benzyl-6-bromo-2-methoxyquinoline (**3**) by single-crystal X-ray diffraction, and no further analyses were performed.

2.1.3. Bedaquilinium fumarate (2). Bedaquiline (30 mg, 0.054 mmol) was mixed with fumaric acid (6.3 mg, 0.054 mmol) dissolved in acetone (1 ml) in a 10 ml scintillation vial. Propyl alcohol (5 ml) was then added and the mixture was allowed to evaporate slowly to obtain large colorless block-shaped crystals that were analyzed by single-crystal and powder X-ray diffraction. ^{1}H NMR (500 MHz, ACN-d_3): δ 8.68 (*d*, *J* = 8.3 Hz, 1H), 8.55 (*s*, 1H), 8.05 (*d*, *J* = 7.2 Hz, 1H), 7.97 (*s*, 1H), 7.88 (*d*, *J* = 7.9 Hz, 1H), 7.66 (*m*, 4H), 7.51 (*t*, *J* = 7.2 Hz, 1H), 7.32 (*m*, 3H), 6.89 (*m*, 3H), 6.32 (*s*, 2H), 5.89 (*s*, 1H), 4.21 (*s*, 3H), 3.02 (*m*, 1H), 2.69 (*m*, 1H), 2.24 (*s*, 7H), 2.09 (*m*, 2H).

2.1.4. Bedaquilinium benzoates 4a and 4b. Bedaquiline (30 mg, 0.054 mmol) was mixed with benzoic acid (6.7 mg, 0.055 mmol). The mixture was dissolved in acetone (2 ml) in a 5 ml scintillation vial and was allowed to evaporate. The 1.17-hydrate **4a** was obtained in the form of colorless rod-shaped crystals (m.p. 127–129 °C). ^{1}H NMR (500 MHz, ACN-d_3): δ 8.75 (*d*, *J* = 8.4 Hz, 1H), 8.67 (*s*, 1H), 8.12 (*d*, *J* = 7.1 Hz, 1H), 7.94 (*d*, *J* = 8.0 Hz, 1H), 7.86 (*m*, 3H), 7.75 (*d*, *J* = 8.0 Hz, 2H), 7.56 (*m*, 4H), 7.38 (*m*, 5H), 6.93 (*m*, 3H), 5.95 (*s*, 1H), 4.26 (*s*, 3H), 3.05 (*m*, 1H), 2.96 (*m*, 1H), 2.25 (*m*, 7H), 1.96 (*m*, 2H). An identical material with the same water content was obtained when crystallization was carried out from 2-propanol instead of acetone.

Bedaquiline (30 mg, 0.054 mmol) was mixed with benzoic acid (6.8 mg, 0.056 mmol) in acetonitrile (10 ml) and was allowed to evaporate slowly. The acetonitrile solvate monohydrate **4b** was obtained in the form of thin colorless needles (m.p. 127–129 °C).

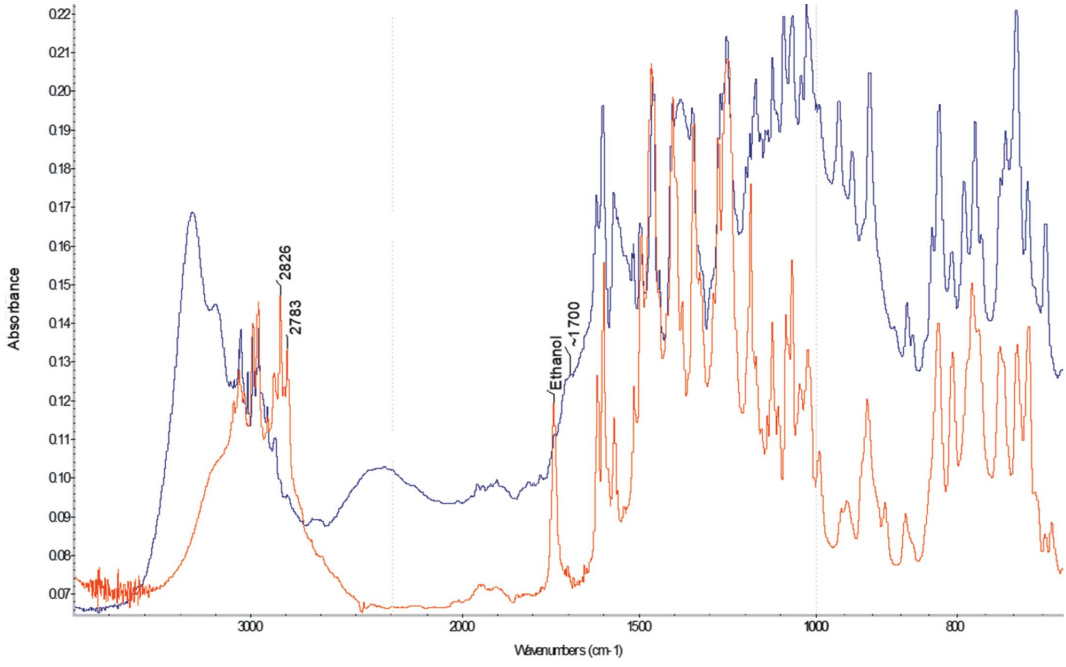


Figure 2
IR spectra of bedaquilinium benzoate (**4a**) (blue) and bedaquiline free base (**1**) (red).

2.2. Refinement

Crystal data, data collection, and structure refinement details are summarized in Table 1. The two benzoate salt structures **4a** and **4b** are isomorphous, differing from each other only in the nature of part of the solvent molecules and some slight shifts to other atoms, and they were refined against a common structural model, with the structure of **4b** being solved by isomorphous replacement from that of **4a**. The atom-naming scheme for the published bedaquiline free base structure (Petit *et al.*, 2007), as deposited in the Cambridge Structural Database (CSD; Groom *et al.*, 2016; refcode KIDWAW), was used for the remeasured 150 K data of free base bedaquiline **1** and was also adopted for the bedaquilinium cations in the two benzoate salts **4a** and **4b**, and fumarate salt **2**.

For powder X-ray data collection and refinement, see the *Experimental* (§2).

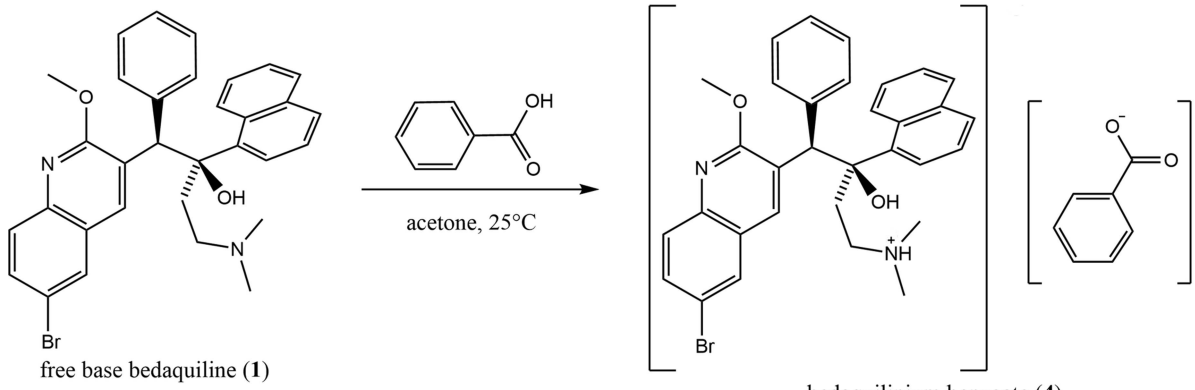
2.2.1. H-atom treatment. C-bound H atoms were added in calculated positions and refined using a riding model. C—H bond distances were constrained to 0.95 Å for aromatic C—H moieties, and to 1.00, 0.99, and 0.98 Å for aliphatic C—H, CH_2, and CH_3 moieties, respectively. Alcohol O—H and ammonium NR_3H N—H bond distances were either freely refined (for **2**) or were constrained to 0.84 and 1.00 Å, respectively. Methyl CH_3 and hydroxy H atoms were allowed to rotate but not to tip to best fit the experimental electron density. The positions of the carboxylate H atoms (in **2**) were refined freely. The positions of the fully occupied water H atoms were refined freely and O—H distances were restrained to 0.84 (2) Å. The H atoms of the partially occupied water molecule in **4a** were initially refined and O—H and H···H distances were restrained to 0.84 (2) and 1.36 (2) Å, respectively, while a damping factor was applied. The position of water atom H6*E* (in **4a**) was further restrained based on hydrogen-bonding considerations, *i.e.* to be hydrogen bonded to the pyridine H atom, with the H···N distance restrained to 2.35 (2) Å. In the final refinement cycles, the damping factor was removed and the H atoms were set to ride on the parent O atom. For all structures, the U_{iso}(H) values were set to a multiple of U_{eq}(C), being 1.5 for CH_3 and OH, and 1.2 for C—H, CH_2, and N—H groups, respectively.

2.2.2. Disorder modeling. In the structure of **4a**, one fully occupied and one partially occupied water molecule are present in the lattice. The occupancy ratio refined to 0.166 (7). In **4b**, the partially occupied water molecule is replaced by an approximately three-quarter-occupied acetonitrile molecule. In the absence of the acetonitrile molecule, the neighboring benzene ring of the benzoate anion tilts slightly to move towards the void left by the absent solvent molecule. The two disordered benzene moieties were restrained to have similar geometries. The U^{ij} components of the anisotropic displacement parameters (ADPs) for disordered atoms closer to each other than 2.0 Å were restrained to be similar. The ADPs of the *ipso* C atoms, which occupy nearly identical positions, were constrained to be identical. Subject to these conditions, the occupancy ratio refined to 0.742 (7):0.258 (7) in favor of the acetonitrile molecule being present.

3. Salt screening and methods

Salt screening is a complex and challenging endeavor involving potentially millions of experiments. For bases like bedaquiline, these experiments can involve up to 50 commonly used salt formers in various ratios, as well as crystallizations from up to 60 different solvents by varying temperature, concentration, agitation, pH, and other factors. Further mixtures of these solvents can be used. Antisolvent crystallization using these solvents is also of interest and introduces even more variables.

For bedaquiline, the first step in screening for additional salts involved recovering bedaquiline free base from the commercially available bedaquilinium fumarate. This was first attempted by deprotonation of the bedaquilinium cation of the fumarate salt using the base sodium ethoxide. However, the alkoxide, when used in excess, proved to be too strong a base and led to fragmentation of the bedaquiline molecule. One of the products of the decomposition reaction was isolated by crystallization and identified, by single-crystal X-ray diffraction, as 3-benzyl-6-bromo-2-methoxyquinoline (**3**) (Fig. 1) and its structure will be described below. The other half of the decomposition reaction was not recovered or identified, but is assumed to be the ketone elimination product



free base bedaquiline (**1**)

bedaquilinium benzoate (**4**)

Figure 3
Synthesis of bedaquilinium benzoate (**4**).

of the remaining bedaquiline fragment, 4-(dimethylamino)-1-(naphthalen-1-yl)butan-2-one. The reaction most likely proceeds through initial deprotonation of all acidic groups by the ethoxide, including the central alcohol of bedaquiline. The tertiary alkoxide thus formed can undergo a reverse Grignard reaction (Zook *et al.*, 1959), under elimination of the ketone and the carbanion of **3**. Using much less basic sodium bicarbonate as the neutralizing agent avoids this decomposition reaction. Bedaquiline free base could be recovered that way from the fumarate salt, following the procedure described by Rombouts *et al.* (2016), thus allowing us to proceed to use the free base in salt screening experiments (Fig. 1).

Because the fumarate, citrate, sulfate, phosphate, and tartrate salts were known, salt formation screening focused on the crystallization of salts such as acetate, benzoate, benzenesulfonate, hydrobromide, succinate (1:1 and 1:2), hydrochloride, tartrate (1:1 and 1:2), lactate, maleate (1:1 and 1:2), malate (1:1 and 1:2), and mesylate. In general, the crystallizations involved mixing stoichiometric amounts of bedaquiline with the acids at either 1:1 or 1:2 molar ratios in acetone, acetonitrile, tetrahydrofuran, and ethyl acetate, either with or without the antisolvents water and hexane. The solvents were evaporated either slowly or rapidly, and materials were typically first screened using polarized-light microscopy (PLM) to ensure that a crystalline material had formed, and that the sample was uniform. Samples that passed the first screening step were submitted for further analysis. Crystals were analyzed by NMR (dissolved in an appropriate solvent) to confirm the presence of both components in the material. In the next step, materials were further screened using IR microspectroscopy, to confirm that the material was indeed a new substance (a salt or a cocrystal), and not just a mixture of the two components. Although some investigators have advanced the theory that Raman spectroscopy is the best method for analysis and determination of salt formation (*e.g.* Kojima *et al.*, 2010), we found IR microspectroscopy had better specificity than Raman microscopy for the bedaquiline free base and salts; therefore, screening materials *via* Raman methods was abandoned. IR microspectroscopy proved to be a superior method to determine the formation of bedaquilinium salts. Materials that passed these screening steps (PLM, NMR, and IR spectroscopy) were then analyzed by powder X-ray diffraction. Rietveld refinement was used to identify known crystal phases. For samples for which suitable crystals could be obtained, single-crystal X-ray diffraction was used to determine the structures of phases not yet structurally described.

Example IR spectra comparing bedaquilinium benzoate and free base bedaquiline are given in Fig. 2; see Fig. 3 for the synthesis. The spectra are distinctly different, indicating transformation of the free base into a material containing both benzoate and bedaquiline fragments. A range of bands in the fingerprint region indicate the presence of a bedaquiline component in both compounds. A shoulder near 1700 cm^{-1} in the bedaquiline benzoate spectrum can be assigned to the C═O stretch of benzoate, confirming the formation of the salt. Further evidence for the formation of a salt, rather than a simple mixture of the two starting materials, is provided by the absence of bands in the range 2830–2760 cm^{-1}. Tertiary amines (of which the free base is one) have a characteristic $N-CH_2$ in-phase stretch that occurs in this range (Colthup *et al.*, 1990). The bands in this range of the free base spectrum are not present in the bedaquiline benzoate spectrum, suggesting the formation of a salt. Note: the free base spectrum contains some spectral features due to ethanol, which was used in the synthesis process.

In the course of our investigations, we had been so far able to determine the single-crystal structures of bedaquilinium fumarate (**2**), the commercially available form of bedaquiline, as well as isolate and characterize two other previously unknown bedaquilinium salts: the mono-benzoate salt, in the form of its 1.17-hydrate (**4a**), and a mixed hydrate acetonitrile solvate (**4b**). Their structures, as well as that of the degradation product from reaction of bedaquilinium fumarate with sodium ethoxide, 3-benzyl-6-bromo-2-methoxyquinoline (**3**), will be described below. The structure of free base bedaquiline (**1**) was re-recorded at 150 K for easier comparison with the low-temperature data of **2**, **4a**, and **4b**. Implications for the larger bedaquiline system will be discussed.

4. Results and discussion

3-Benzyl-6-bromo-2-methoxyquinoline (**3**), the solvolysis product from reaction of bedaquiline with sodium ethoxide, lacks an easy-to-identify NMR signature and was identified by single-crystal X-ray diffraction. It crystallized from acetonitrile in the orthorhombic and chiral space group $P2_12_12_1$ (Fig. 4). The starting bedaquilinium fumarate is a chiral compound and an enantiopure sample was used. This chiral information and all chiral centers are, however, lost in the degradation reaction to 3-benzyl-6-bromo-2-methoxyquinoline (**3**). In the solid state, the molecule does, however, exhibit chirality, and the crystal analyzed was found to be enantiopure, with a Flack parameter of −0.011 (3). In solution, the

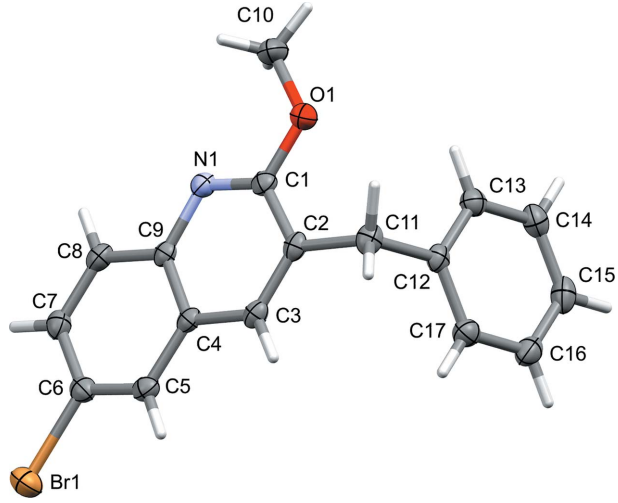


Figure 4
The structure of decomposition product **3** (50% probability displacement ellipsoids).

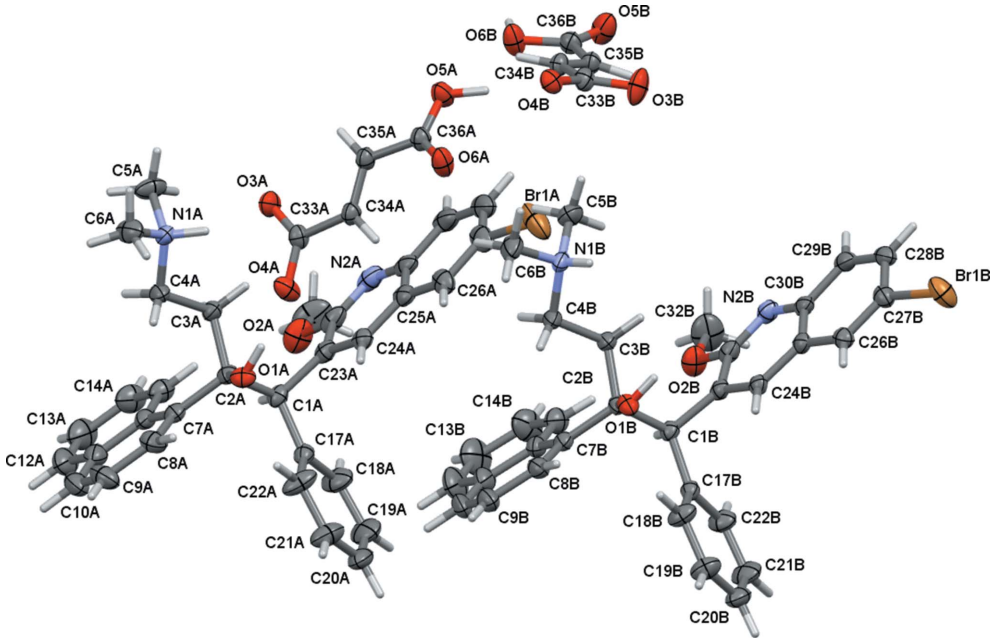


Figure 5
Single-crystal structure of fumarate salt **2** (50% probability displacement ellipsoids).

material is expected to be a rapidly interconverting racemic mixture, as simple rotation of the benzyl group to the other side of the mean plane of the molecule creates the inversion-related enantiomer. Molecules of **3** are divided into two planar fragments: the benzyl group and the 6-bromo-2-methoxyquinoline moiety. Both fragments are close to ideally flat, with r.m.s. deviations from planarity of only 0.0052 and 0.0194 Å, respectively. The methoxy group is thus ideally coplanar with the remainder of the bromoquinoline fragment. It points away from the benzyl CH_2 group to avoid steric interactions. The torsion angle between the two mean planes is 73.01 (4)°.

The structures of the three bedaquilinium salts, *i.e.* fumarate **2**, and benzoates **4a** and **4b**, are substantially more complicated (Figs. 5 and 6), but they share some commonalities. In all three salt structures, the bedaquilinium cation is singly protonated at the dimethylamino fragment, with 1:1 anion-to-cation ratios. In the structure of **2**, the fumarate anions remain singly protonated hydrofumarate(1−) anions, thus being monoanionic, as are the benzoate anions. The quinoline N atoms remain unprotonated, even though there are additional acidic protons available in the structure of **2**. At first glance, this might be surprising, since many pyridinium salts of both benzoic and fumaric acids have been reported [973 pyridinium benzoate derivatives and 44 pyridinium fumarate salts are reported in the CSD (Groom *et al.*, 2016), accessed August 2020]. The behaviour is, however, in agreement with the pK_a

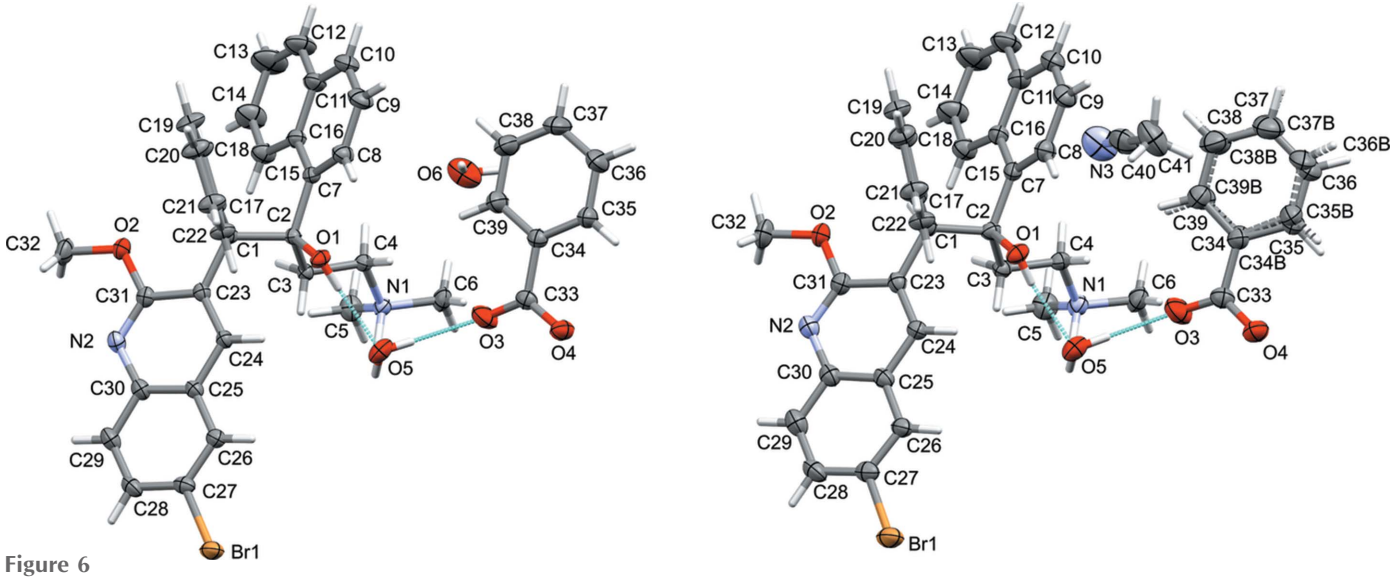


Figure 6
Single-crystal structures of benzoate salts of **4a** (left) and **4b** (right) (50% probability displacement ellipsoids). Hydrogen bonds are shown as turquoise dashed lines.

values of the acids and with the reduced basicity of the quinoline N atom of bedaquiline, compared to ordinary pyridine. The first pK_a of fumaric acid is 3.053, the second is 4.494, and that of benzoic acid is 4.202 (Martell & Smith, 1976), which are easily sufficient to protonate the amine moiety of bedaquiline [the pK_a of trialkylammonium ions are around 10–11 (Bioquest, 2020)]. The pK_a of the conjugated acid of bedaquiline protonated at the quinoline N atom is not reported but can be estimated from the known values for quinoline, pyridine, and 2-methoxypyridine, which are 4.9, 5.23, and 3.06, respectively (Bioquest, 2020). Methoxy substitution in the 2-position to the N atom substantially reduces the basicity of the N atom (the pK_a of the conjugated acid drops by 2.17 between pyridine and 2-methoxypyridine). Assuming other effects to be negligible yields a pK_a value of 2.73 for 2-methoxyquinolinium. The quinoline N atom of bedaquiline is thus not basic enough to be protonated by medium-strength acids, such as benzoic or fumaric acid, in agreement with the findings from the crystal structures for **2**, **4a**, and **4b**. Stronger acids, such as mineral acids or maleic acid (first pK_a is 1.94; Bioquest, 2020), might be able to double protonate bedaquiline if used in sufficient excess. Experiments in this direction are ongoing in our laboratories.

All three salts do crystallize in the chiral monoclinic space group $P2_1$. The core of the bedaquilinium cation in the three structures is formed by the ethylene moiety of atoms C1 and C2, from which the four major substituents radiate off: the phenyl ring and the bromo(methoxy)quinoline group from C1, and the naphthyl and (dimethylamino)ethyl fragments from C2. The hydroxy group is also attached to C2, while C1 also carries a single H atom. Atoms C1 and C2 are also the chiral centers of the bedaquilinium cation, which were modeled to have *R* and *S* chiralities, respectively, in agreement with the reported absolute structure for free base bedaquiline (Petit *et al.*, 2007). The Flack parameters refined to −0.014 (1) for **2**, and to 0.006 (3) and 0.004 (8) for **4a** and **4b**, respectively, confirming that the crystals were enantiopure.

The arrangement of anions and cations, and packing interactions, however, vary substantially between the fumarate and the two benzoate salts. The fumarate salt crystallized in an anhydrous and unsolvated form. Two crystallographically unique ion pairs are present in the lattice (*i.e.* $Z' = 2$ for compound **2**). The structure obtained agrees with the reported powder patterns of commercially available bedaquilinium fumarate (see Fig. 7 for a Rietveld refinement plot).

The two newly isolated benzoate salts are distinctly different from **2**, both being solvates, but they are very similar to each other, and are indeed isomorphous (the acetonitrile solvate was solved by isomorphous replacement from the hydrate). Both structures feature one tightly bound water molecule (atom O5). A second interstitial site does, however, differ between the two phases. In **4a**, it is occupied by a second water molecule, which is partially occupied. In **4b**, on the other hand, this site is partially occupied by a disordered acetonitrile molecule, which in turn induces disorder in the phenyl ring of the benzoate anion [see *Refinement* (§2.2.2) for disorder details].

The ethane backbone of the bedaquiline core gives the cations a three-dimensional (3D) shape, but the individual

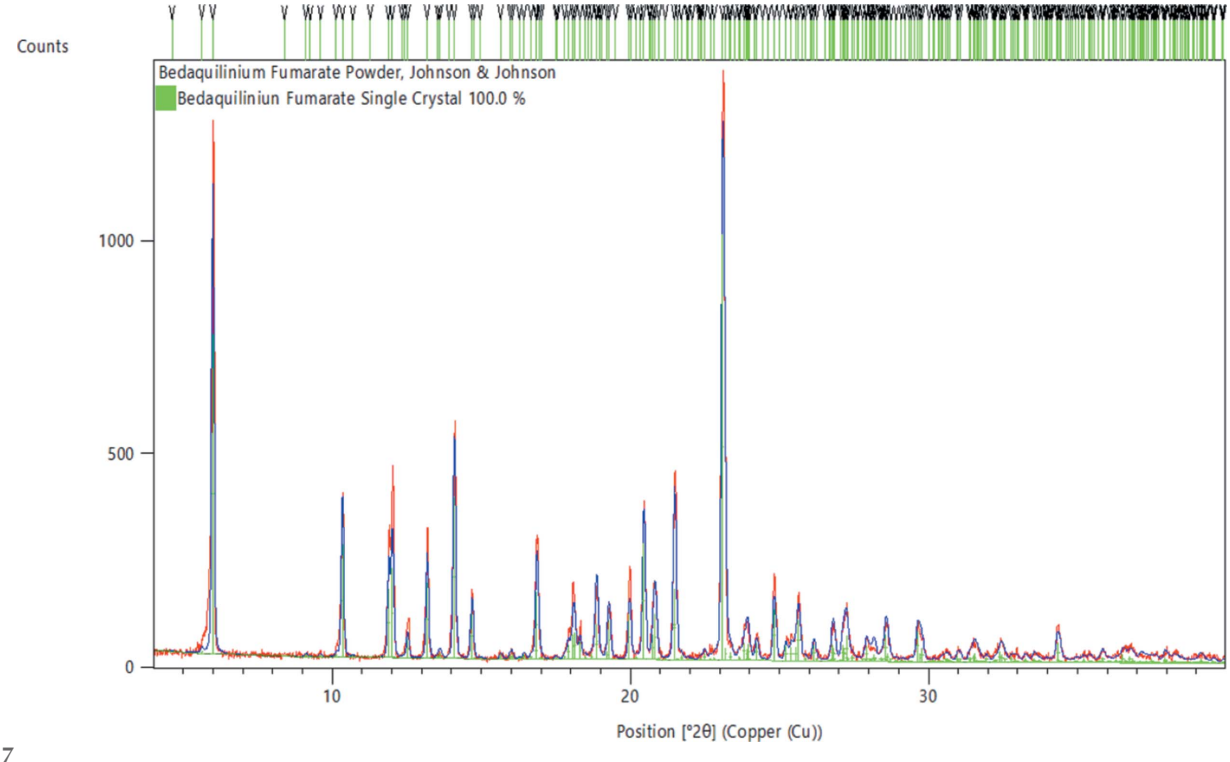


Figure 7
Powder XRD pattern (ambient temperature) of bedaquilinium fumarate (Johnson & Johnson) with a Rietveld refinement fit against the single-crystal structure of **2**. The room-temperature unit-cell parameters refined to a = 16.5879 (2), b = 10.4952 (8), c = 20.183 (2) Å, β = 109.238 (2)° and V = 3317.4 (4) Å^3. Rietveld fits for **4a** and **4b** are given in the supporting information.

Table 2
Selected torsion angles (°) for free base bedaquiline **1**, fumarate salt **2**, and benzoate salts **4a** and **4b**.

	1[a,b]	**2**[b]	**4a**[a]	**4b**[a]
τ plane 1 *versus* plane 2[c]	73.29 (7), 81.37 (7)	85.33 (9), 86.02 (8)	77.31 (6)	76.2 (1)
τ plane 2 *versus* plane 3[c]	86.13 (8) 76.95 (7)	89.7 (1), 89.74 (9)	45.62 (6)	44.2 (1)
τ plane 1 *versus* plane 3[c]	14.0 (1), 8.16 (9)	8.71 (3), 16.67 (4)	37.50 (6)	36.7 (1)
τ C1—C2—C3—C4	175.7 (3), 177.0 (3)	−63.8 (3), 174.92 (19)	166.84 (14)	165.1 (3)
τ C2—C3—C4—N1	58.7 (4), 63.0 (4)	137.2 (2), 133.7 (2)	164.04 (15)	165.5 (3)
τ C17—C1—C2—C3	169.0 (3), 178.8 (3)	169.7 (2), 171.38 (19)	174.44 (14)	174.1 (3)

Notes: (*a*) measured at 150 K; (*b*) Z' = 2; (*c*) plane 1 = 6-bromo-2-methoxyquinoline, plane 2 = phenyl, and plane 3 = naphthyl.

Table 3
Hydrogen-bond geometry (Å, °) for **1**.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···N1	0.84	1.94	2.696 (4)	150
C1—H1···O2	1.00	2.24	2.763 (4)	111
O3—H3O···N3	0.84	1.93	2.685 (4)	149
C33—H33···O4	1.00	2.23	2.773 (4)	112

Table 4
Hydrogen-bond geometry (Å, °) for **2**.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1*AB*···O4*A*	0.86 (4)	1.88 (4)	2.699 (3)	159 (4)
O5*A*—H5*A*···O4*B*	1.04 (4)	1.58 (4)	2.603 (3)	169 (4)
N1*A*—H1*A*N···O3*A*	0.98 (4)	1.68 (4)	2.641 (3)	163 (3)
C1*A*—H1*A*···O2*A*	1.00	2.23	2.767 (3)	112
C3*A*—H3*AB*···Br1*B*^{i}	0.99	3.01	3.850 (3)	143
C5*A*—H5*AA*···O1*A*^{ii}	0.98	2.58	3.501 (3)	157
C5*A*—H5*AC*···Br1*B*^{i}	0.98	2.82	3.612 (3)	139
C26*A*—H26*A*···O5*B*^{iii}	0.95	2.54	3.459 (4)	163
C34*A*—H34*A*···O5*B*^{iii}	0.95	2.52	3.379 (3)	151
O1*B*—H1*B*···O4*B*^{iv}	0.89 (4)	1.88 (4)	2.741 (3)	160 (4)
O6*B*—H6*B*···O4*A*^{v}	0.86 (5)	1.80 (5)	2.625 (3)	159 (4)
N1*B*—H1*B*N···O3*B*^{iv}	0.85 (4)	1.83 (4)	2.632 (3)	158 (4)
C5*B*—H5*BA*···O1*B*^{vi}	0.98	2.42	3.337 (4)	156
C5*B*—H5*BB*···Br1*A*	0.98	2.88	3.468 (3)	119
C5*B*—H5*BB*···O6*A*	0.98	2.42	3.287 (4)	148

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

aromatic fragments remain planar. Similar to **3**, the 6-bromo-2-methoxyquinoline moiety is planar, with the methoxy group pointing away from the core of the cation. The r.m.s. deviations from planarity are 0.1127 and 0.1363 for the two cations in **2** (Z' = 2), 0.1019 Å in **4a**, and 0.0922 Å in **4b**.

The ethane backbone and the malleable ethylamine fragment gives the bedaquilinium cations a high degree of conformational flexibility. Differing packing arrangements, induced by the presence (or absence) of varying anions and solvent molecules, led to a landscape of conformations observed for the cations in **2**, **4a**, and **4b**, as well as free base bedaquiline **1**. The dihedral angles between the mean planes of the 6-bromo-2-methoxy-quinoline fragment (plane 1), the phenyl ring (plane 2), and the naphthyl group (plane 3), as well as the torsion angles along the ethane backbone and the ethylamine fragment, are given in Table 2. Besides the obvious similarities between the values for isomorphous **4a** and **4b**, no general trend is observed. The conformations vary not only between the four structures, but even between independent molecules within the same structure (both free base **1** and fumarate **2** are Z' = 2 structures). The two C1—C2—C3—C4 torsion angles in fumarate salt **2**, for example, are −63.8 (3) and 174.92 (19)°, which are distinctly different from each other. However, some similarities can be observed: the interplanar angles between the phenyl and 6-bromo-2-methoxyquinoline planes are between 70 and 90° in all structures, and the torsion angles involving the ethane backbone and the *ipso* phenyl atom (C17—C1—C2—C3) are close to antiperiplanar ('*trans*') in all the compounds. No other similarities common to all four structures can be found and the overall trend is one of pronounced flexibility and variability.

While the geometries and conformations in the four bedaquiline structures do not follow any general trend, there are some differences between the geometries of free base bedaquiline and its salts that can be rationalized. Directional interactions that differ between the free base and the salts play a major role. In bedaquiline, only one actual hydrogen bond is present, and this is an intramolecular O—H···N hydrogen bond (Table 3). It induces the amine N atoms to turn towards the hydroxy group, thus enforcing a *gauche* geometry of the (dimethylamino)ethyl group (see torsion angle C2—C3—C4—N1 in Table 2). Intermolecular interactions between molecules in **1** are limited to weaker and less directional interactions, specifically Br···Br interactions and π-stacking, which had been discussed in detail by Petit *et al.* (2007). In the fumarate and benzoate salts **2** and **4**, the opposite is observed. The amine moiety, being protonated, is unable to act as the acceptor for an O—H···N hydrogen bond, and it takes up the role of a hydrogen-bond donor towards the benzoate or fumarate anions. This releases the amine from the *gauche* conformation enforced by the intramolecular O—H···N hydrogen bond in **1**, and the ethylamine instead extends into a more relaxed conformation, approaching *anti* in **4** and close to eclipsed in **2** (see torsion angle C2—C3—C4—N1 in Table 2). The hydroxy groups in the salts are freed up to also form intermolecular hydrogen bonds. In **4**, they hydrogen bond with the fully occupied water molecule, and in **2** with a fumarate carboxylate group. The water molecule in **4** in turn extends hydrogen bonds to the O atoms of two neighboring benzoate anions, and the fumarate anions in **2** are involved in a series of hydrogen bonds among each other, forming an infinite chain of hydrogen-bonded anions along the *b*-axis direction (Fig. 8). Thus, one intramolecular hydrogen bond in free base bedaquiline **1** is converted into a whole series of strong intermolecular hydrogen bonds (Tables 4, 5 and 6), creating for the

Table 5
Hydrogen-bond geometry (Å, °) for **4a**.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O5	0.84	1.87	2.681 (2)	164
O5—H5*D*···O4^{i}	0.90 (4)	1.85 (4)	2.724 (2)	163 (3)
O5—H5*E*···O3	0.77 (3)	1.96 (3)	2.724 (2)	172 (4)
O6—H6*E*···N2	0.87	2.38	3.148 (13)	147
N1—H1···O4^{i}	1.00	1.64	2.643 (2)	178
C5—H5*C*···Br1^{ii}	0.98	3.09	3.910 (2)	142
C6—H6*A*···O3	0.98	2.53	3.465 (3)	161
C6—H6*B*···O6^{iii}	0.98	2.28	3.249 (13)	169
C28—H28···O5^{ii}	0.95	2.59	3.421 (3)	146

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

Table 6
Hydrogen-bond geometry (Å, °) for **4b**.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O5	0.77	1.94	2.691 (4)	163
O5—H5*D*···O4^{i}	0.86 (3)	1.89 (3)	2.738 (4)	168 (6)
O5—H5*E*···O3	0.82 (3)	1.92 (3)	2.734 (4)	172 (6)
N1—H1···O4^{i}	1.00	1.62	2.619 (4)	178
C5—H5*C*···Br1^{ii}	0.98	3.13	3.964 (4)	144
C6—H6*A*···O3	0.98	2.63	3.562 (6)	159
C28—H28···O5^{ii}	0.95	2.66	3.498 (5)	148

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

salts a much stronger cohesion between neighboring structural entities than what was observed in the free base structure. Weaker interactions that dominate for bedaquiline, such as Br···Br halogen bonds and π-stacking, are not observed for the salts, but the strong hydrogen bonds are augmented by a range of other not-as-strong directional interactions, such as C—H···O and C—H···Br hydrogen bonds (see hydrogen-bond Tables 4, 5, and 6 for a full listing).

In the acetonitrile solvate **4b**, an H atom of the naphthyl group interacts in a C—H···N hydrogen-like bond with the

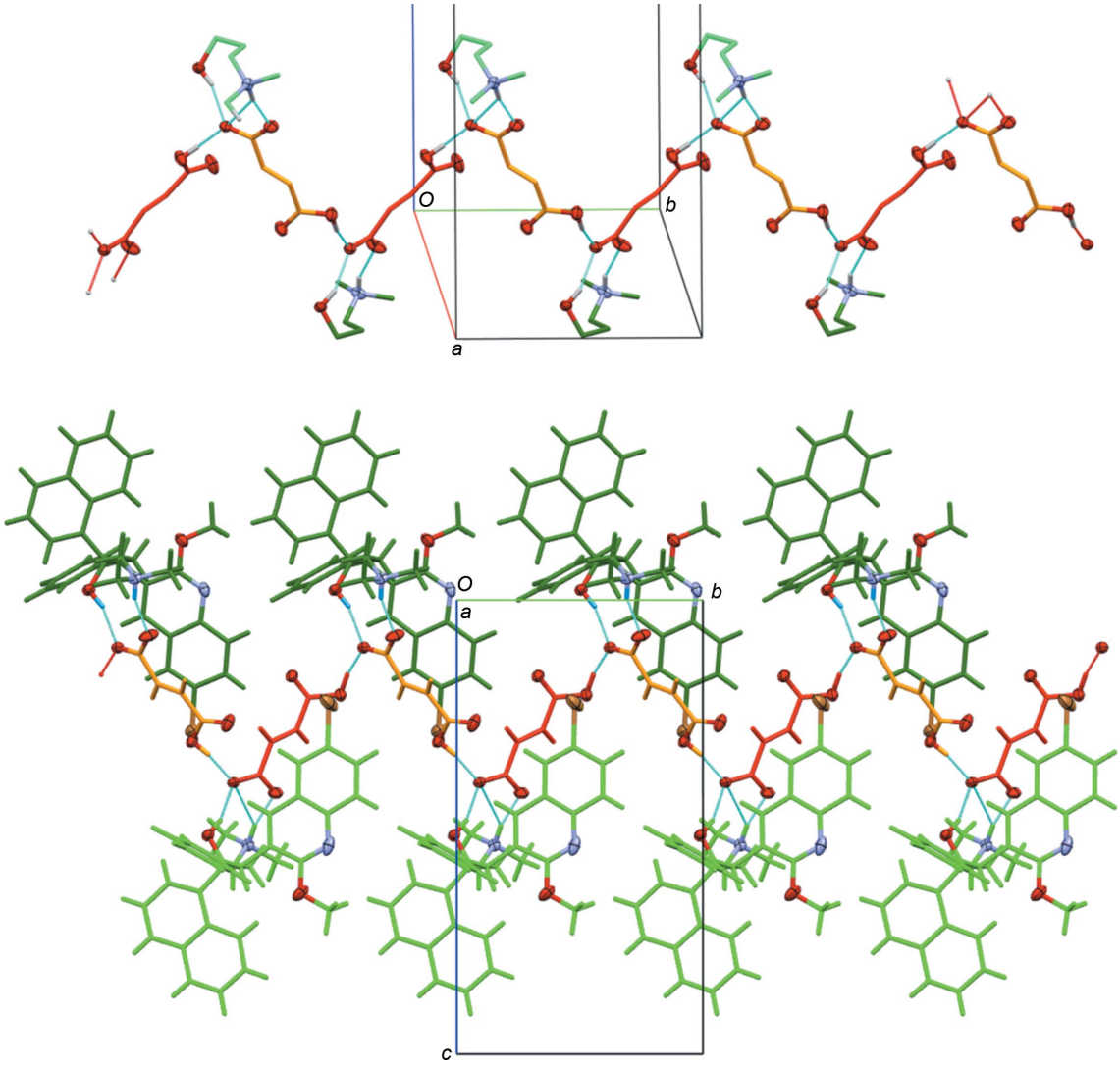


Figure 8
Hydrogen-bonding pattern in **2**. (Top) Chains along *b* created by hydrofumarate anions. For clarity, only the 3-dimethylazaniumyl-1-hydroxypropyl fragments of the cations are shown, and H atoms not involved in hydrogen bonds have been omitted. (Bottom) Segment of an entire ribbon, including full cations. Color coding: O, N, and Br atoms are shown with 50% probability displacement ellipsoids, with all other atoms in capped-stick mode and color coded by the symmetry equivalence of their anion and cation (*A* and *B* cations in light and dark green, and *A* and *B* anions in orange and red). Hydrogen bonds are shown as turquoise and red dashed lines.

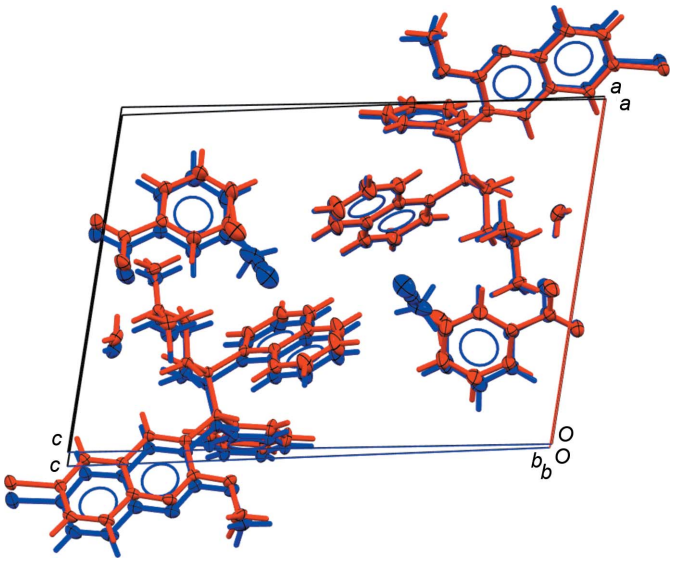


Figure 9
Least-squares overlay of the structures of **4a** (red) and **4b** (blue) using the non-H atoms for one of the two ion pairs of the asymmetric unit (r.m.s. fit value = 0.152), ignoring water and solvent molecules, and disorder. When extending the fit to the atoms of both ion pairs of the unit cell, the r.m.s. value increases to 0.195, showing the lattice mismatch between **4a** and **4b**.

solvent molecule, when present. In its absence, the phenyl ring of the benzoate anion relaxes towards the void created, inducing disorder for the anion [see *Refinement* (§2.2.2) for details]. For the 1.17-hydrate **4a**, the partially occupied water molecule is hydrogen bonded to the pyridine N atom and acts as an acceptor for a C—H···O interaction. There is, however, no second possible hydrogen-bond acceptor near the partially occupied water molecule, and the second water H atom is not involved in any recognizable interaction. This is energetically unfavorable, which helps to explain why this position is occupied less than 20% of the time [the refined value is 16.6 (7)%], while the other solvent water molecule, tightly hydrogen bonded on all sides, is fully occupied. Lack of space appears to be no issue, as the larger acetonitrile molecule in **4b** has a higher occupancy of around three-quarters [refined value 74.2 (7)%]. Kinetic factors during crystal growth (a lack of water molecules during crystallization from mostly dry solvents for **4a**, but no lack of acetonitrile molecules for **4b**) seem to dominate. The presence or absence of either water or acetonitrile in this void does not appear to hinder continuation of crystal growth, while this cannot be said for the tightly incorporated O5 water molecule present in both structures, which appears to be essential for the formation of this structure. The larger size of acetonitrile *versus* water, and the low prevalence of the second water molecule in **4a**, lead to a slightly larger unit-cell volume for **4b** compared to **4a**, *i.e.* 1773.13 (19) *versus* 1729.63 (12) Å^3. The visually most obvious difference between the two structures is the difference in the β angle, which is expanded by slightly more than 1° in **4b**, leading to a significant offset between atoms in the two structures when the unit cells are overlaid (Fig. 9). The same is evident when comparing the powder patterns of **4a** and **4b** simulated from the 150 K single-crystal data, which are distinctively different (see supporting information).

Observations based on powder XRD data indicate that the samples of **4b** quickly desolvate, even under ambient conditions. The powder patterns of **4b** more closely match the parameters of hydrate **4a** than would be expected for acetonitrile solvate **4b**, even if the data were collected on samples exposed to air only for a few minutes prior to data collection (Table 7). Rietveld refinements of samples of **4b** yield β angles that match those of **4a** (at both room temperature and 150 K), and the room-temperature volume of **4b** is actually slightly smaller than that of **4a**, in agreement with the assumption that all the acetonitrile in the structure of **4b** is lost when exposed to the atmosphere, while the solvent water molecules of **4a**, being hydrogen bound, do remain in the lattice under these conditions. These observations will be further investigated in a more detailed upcoming study, focusing on a larger series of bedaquilinium salts, including their solvation properties, thermal stability, and responses to ambient moisture.

The structure of **2**, on the other hand, is solvent free. Like **4**, the main intermolecular interactions are strong hydrogen bonds, already briefly discussed, with the bedaquilinium cation always acting as a hydrogen-bond donor, and the hydrofumarate anions acting as both H-atom acceptors and donors. The quinoline N atom does not act as an acceptor for a hydrogen bond, in agreement with its reduced basicity, already discussed. Originating from the cation are N—H···O hydrogen bonds, formed by the ammonium cations, and O—H···O hydrogen bonds, originating from the alcohol moieties. The N—H···O and O—H···O hydrogen bonds from one cation are towards the two O atoms of

Table 7
150 K and room-temperature (RT) unit-cell dimensions for **1**, **2**, **4a**, and **4b**.

RT data were obtained from powder XRD patterns *via* Rietveld refinement (see supporting information for Rietveld plots).

	1, 150 K	**1**, RT	**2**, 150 K	**2**, RT
a (Å)	11.1584 (8)	11.230 (1)	16.4556 (6)	16.5879 (2)
b (Å)	13.6425 (14)	13.766 (1)	10.3205 (3)	10.4952 (8)
c (Å)	36.061 (4)	36.455 (3)	20.1636 (8)	20.183 (2)
β (°)	90	90	109.1832 (15)	109.238 (2)
V (Å^3)	5489.5 (9)	5636 (1)	3234.2 (2)	3317.4 (4)
ρ (Mg m^{-3})	1.344	1.309	1.379	1.344
R_{wp} (%)		19.99		20.48
R_{exp} (%)		15.67		13.27
S		1.27		1.54

	4a, 150 K	**4a**, RT	**4b**, 150 K	**4b**, RT[a]	
a (Å)	12.6384 (5)	12.7267 (4)	12.8661 (8)	12.720 (1)	12.7242 (8)
b (Å)	7.9259 (3)	8.0157 (3)	8.0386 (5)	8.0094 (5)	8.0116 (6)
c (Å)	17.5249 (8)	17.6438 (7)	17.4704 (10)	17.626 (2)	17.632 (1)
β (°)	99.8450 (17)	99.7928 (6)	101.093 (3)	99.836 (2)	99.813 (1)
V (Å^3)	1729.63 (12)	1773.7 (1)	1773.13 (19)	1769.4 (3)	1771.2 (2)
ρ (Mg m^{-3})	1.342	1.309	1.360	1.311	1.361
R_{wp} (%)		5.57		30.65	28.65
R_{exp} (%)		10.78		10.50	14.26
S		1.94		2.12	2.01

Note: (*a*) data from two independent samples and Rietveld refinements.

the same fully deprotonated carboxylate group, yielding an $R_2^2(10)$ hydrogen-bonding graph-set motif. This motif is identical for the two crystallographically independent ion pairs in **2**. The remaining strong hydrogen bonds are between the hydrofumarate anions. The hydrogen-bond donors are the carboxylic acid groups of each hydrofumarate anion, while the hydrogen-bond acceptor is always the O atom also hydrogen bonded to the bedaquilinium alcohol group, thus tying fumarate anions together in a head-to-tail fashion, forming infinite chains that extend along the *b*-axis direction (Fig. 8). The individual hydrogen-bonding motifs for these hydrogen bonds are $D(2)$. The hydrofumarate anion chains bridge between bedaquilinium cations connecting anions and cations into ribbons that extend along the *b*-axis direction.

These strong hydrogen bonds in **2** are again augmented by a series of other not-as-strong directional interactions, such as C—H···O and C—H···Br hydrogen bonds (Table 4) that interconnect between ribbons to create a fully 3D network and structure. Segments of neighboring ribbons do also interdigitate with each other, further stabilizing the overall structure.

The arrangement of anions and cations in **4a** and **4b**, and their connection *via* hydrogen-bonding interactions, follows a similar pattern to that in **2**, but it is augmented by the solvate water molecules, which play a similar role as the protonated fumarate carboxylic acid groups do in **2** in connecting anions and cations with each other into infinite ribbons (Fig. 10). Bedaquilinium cations and water molecules act as hydrogen-

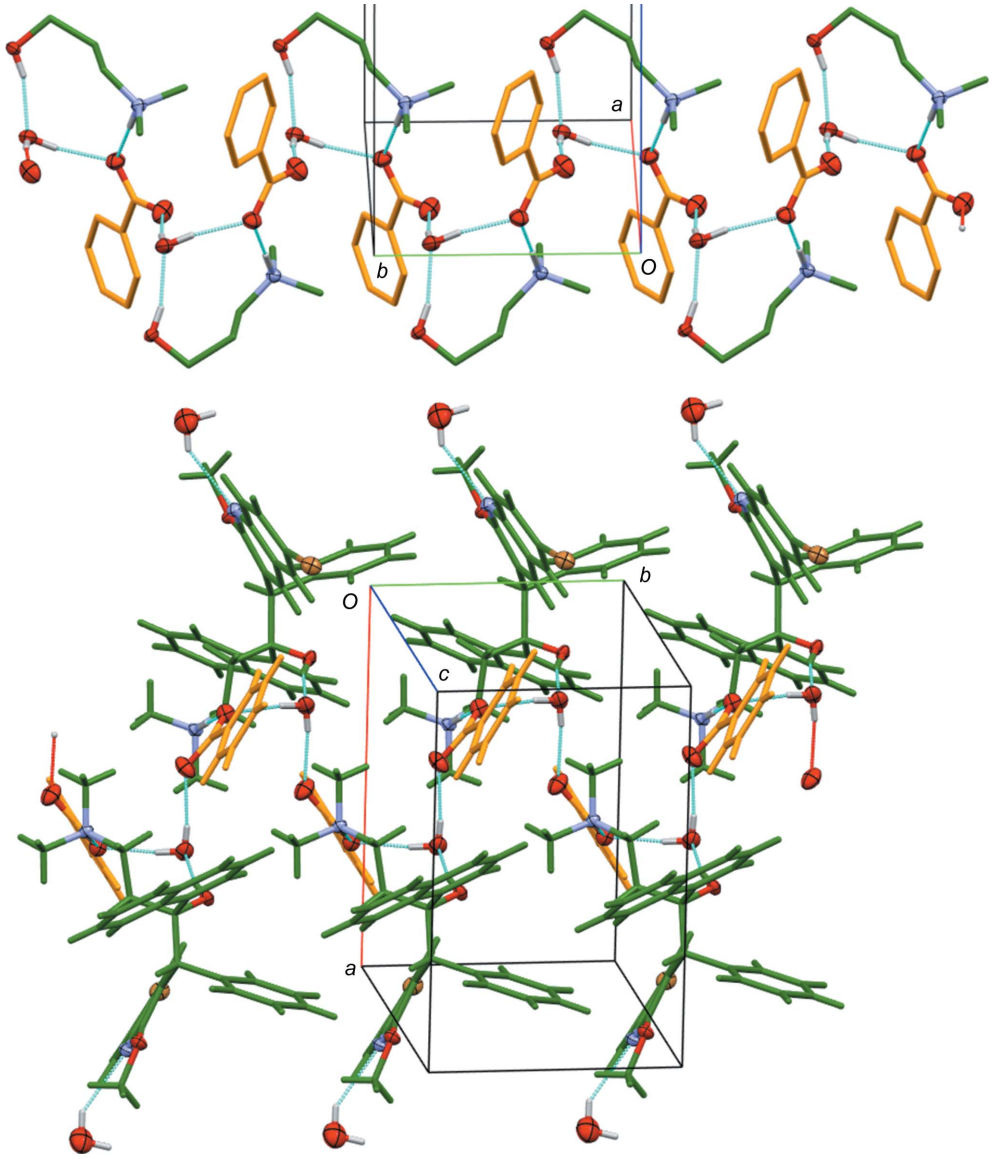


Figure 10
Hydrogen-bonding pattern in **4a**. (Top) Chains along *b* created by benzoate anions and water molecules. For clarity, only the 3-dimethylazaniumyl-1-hydroxypropyl fragments of the cations are shown, and H atoms not involved in hydrogen bonds have been omitted. (Bottom) Segment of an entire ribbon, including cations and partially occupied water molecules. Color coding: O, N, and Br atoms are shown with 50% probability displacement ellipsoids, and all other atoms are in capped-stick mode and color coded by the symmetry equivalence of their anion and cation (cations in dark green and benzoate anions in orange). Hydrogen bonds are shown as turquoise and red dashed lines.

bond donors and the benzoate anions act as hydrogen-bond acceptors. The N—H···O hydrogen bond from the cation is towards one O atom of the benzoate carboxylate group and the O—H···O hydrogen bond is towards the water molecule, which in turn is hydrogen bonded to the same benzoate O atom as the ammonium fragment. The $R_2^2(10)$ graph-set motif in **2** is thus converted in **4** into an $R_3^3(10)$ motif, but otherwise fulfils the same function as in **2**, connecting the hydroxy and ammonium segments of one cation to the same carboxylate group. The fully occupied water molecules in **4** assume the role of the carboxylic acid groups in **2**, acting as bridges between anions [$C_2^2(6)$ hydrogen-bonding motif], creating infinite benzoate–water chains that extend along the *b*-axis direction. Wrapped around these chains, and connected to them *via* O—H···O and N—H···O hydrogen bonds, are the bedaquiline cations, thus creating wider ribbons of cations, anions, and solvent water molecules (Fig. 10), emulating the pattern already observed for **2**. The partially occupied water molecules are located on the outer rim of the ribbon, hydrogen bonded to the quinoline N atom but not bridging or connecting between any structural entities.

The strong intermolecular interactions present in **4a** and **4b** do compensate for the presence of the only partially or not at all filled voids present, and the packing efficiency for the salts of **4** is comparable to that of free base bedaquiline. Indeed, the density for the 1.17-hydrate is, at 1.342 Mg m^{-3}, virtually identical to that of the free base of 1.344 Mg m^{-3} (both measured at 150 K), and the acetonitrile solvate is, at 1.360 Mg m^{-3}, even denser than the solvent-free base. The best packing efficiency is, however, observed for fumarate salt **2**, featuring a multitude of strong hydrogen bonds, and having neither incorporated solvent molecules nor unoccupied void space. Its density, at 150 K, is 1.379 Mg m^{-3} (Table 7).

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supporting information

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Crystal structures of salts of bedaquiline

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Computing details

For all structures, data collection: *APEX3* (Bruker, 2019); cell refinement: *SAINT* (Bruker, 2019); data reduction: *SAINT* (Bruker, 2019); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015) and shelXle (Hübschle *et al.*, 2011); molecular graphics: *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *publCIF* (Westrip, 2010).

1-(6-Bromo-2-methoxyquinolin-3-yl)-4-(dimethylamino)- 2-(naphthalen-1-yl)-1-phenylbutan-2-ol (1)

Crystal data

$C_{32}H_{31}BrN_2O_2$
M_r = 555.50
Orthorhombic, $P2_12_12_1$
a = 11.1584 (8) Å
b = 13.6425 (14) Å
c = 36.061 (4) Å
V = 5489.5 (9) Å^3
Z = 8
$F(000)$ = 2304
D_x = 1.344 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9979 reflections
θ = 2.4–28.7°
μ = 1.53 mm^{-1}
T = 150 K
Plate, colourless
0.21 × 0.13 × 0.05 mm

Data collection

Bruker D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD)
Radiation source: fine focus sealed tube X-ray source
Triumph curved graphite crystal monochromator
Detector resolution: 7.4074 pixels mm^{-1}
ω and phi scans
Absorption correction: multi-scan (SADABS2016; Krause *et al.*, 2015)
T_{min} = 0.603, T_{max} = 0.747
66520 measured reflections
17893 independent reflections
12296 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 33.2°, θ_{min} = 2.3°
h = −14→17
k = −20→17
l = −55→52

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.111
S = 1.03
17893 reflections
675 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0298P)^2 + 2.4789P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.58$ e Å^{-3}

Absolute structure: Flack *x* determined using 4397 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: 0.034 (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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	−0.22418 (4)	0.59893 (4)	0.93403 (2)	0.04766 (11)
O1	0.3238 (2)	0.60084 (19)	0.79619 (6)	0.0300 (5)
H1O	0.296203	0.640769	0.780645	0.045*
O2	0.4828 (2)	0.72509 (17)	0.90856 (6)	0.0311 (5)
N1	0.3008 (3)	0.7681 (3)	0.75701 (9)	0.0374 (7)
N2	0.2968 (2)	0.7233 (2)	0.93579 (8)	0.0289 (6)
C1	0.4337 (3)	0.5908 (2)	0.85326 (9)	0.0249 (6)
H1	0.511656	0.613215	0.863960	0.030*
C2	0.4218 (3)	0.6444 (2)	0.81522 (9)	0.0261 (6)
C3	0.3911 (3)	0.7541 (2)	0.81996 (10)	0.0287 (7)
H3A	0.311884	0.760086	0.832083	0.034*
H3B	0.451320	0.784829	0.836451	0.034*
C4	0.3885 (3)	0.8096 (3)	0.78309 (10)	0.0330 (7)
H4A	0.368347	0.879127	0.787832	0.040*
H4B	0.469185	0.807406	0.771700	0.040*
C5	0.1807 (4)	0.8058 (4)	0.76442 (14)	0.0549 (12)
H5A	0.181362	0.877472	0.762872	0.082*
H5B	0.155422	0.785688	0.789317	0.082*
H5C	0.124585	0.779366	0.746042	0.082*
C6	0.3352 (5)	0.7891 (4)	0.71826 (12)	0.0540 (12)
H6A	0.274637	0.761942	0.701453	0.081*
H6B	0.413095	0.759038	0.712972	0.081*
H6C	0.340535	0.860126	0.714641	0.081*
C7	0.5377 (3)	0.6349 (2)	0.79184 (10)	0.0292 (7)
C8	0.5303 (3)	0.5879 (3)	0.75814 (10)	0.0358 (8)
H8	0.459085	0.553241	0.752149	0.043*
C9	0.6254 (4)	0.5894 (3)	0.73209 (11)	0.0437 (9)
H9	0.617529	0.555399	0.709222	0.052*
C10	0.7269 (4)	0.6388 (3)	0.73966 (12)	0.0479 (10)
H10	0.788893	0.641601	0.721614	0.058*
C11	0.7426 (3)	0.6867 (3)	0.77407 (13)	0.0431 (10)
C12	0.8498 (4)	0.7363 (4)	0.78199 (17)	0.0594 (14)
H12	0.910315	0.740322	0.763505	0.071*
C13	0.8689 (4)	0.7790 (4)	0.81573 (18)	0.0624 (14)

H13	0.942439	0.811591	0.820635	0.075*
C14	0.7811 (4)	0.7748 (3)	0.84279 (15)	0.0529 (11)
H14	0.795439	0.803479	0.866381	0.064*
C15	0.6736 (4)	0.7297 (3)	0.83590 (12)	0.0377 (8)
H15	0.613991	0.729114	0.854720	0.045*
C16	0.6489 (3)	0.6837 (3)	0.80140 (11)	0.0338 (8)
C17	0.4406 (3)	0.4791 (2)	0.85227 (9)	0.0271 (6)
C18	0.5242 (3)	0.4342 (3)	0.87547 (11)	0.0361 (8)
H18	0.578525	0.473635	0.889212	0.043*
C19	0.5296 (4)	0.3329 (3)	0.87886 (13)	0.0455 (10)
H19	0.586943	0.304062	0.894989	0.055*
C20	0.4523 (4)	0.2738 (3)	0.85897 (14)	0.0468 (10)
H20	0.455743	0.204480	0.861264	0.056*
C21	0.3706 (4)	0.3174 (3)	0.83584 (13)	0.0453 (10)
H21	0.317076	0.277449	0.821984	0.054*
C22	0.3643 (4)	0.4183 (3)	0.83226 (12)	0.0374 (8)
H22	0.307030	0.446401	0.815903	0.045*
C23	0.3375 (3)	0.6233 (2)	0.88094 (9)	0.0255 (6)
C24	0.2215 (3)	0.5925 (2)	0.88015 (9)	0.0266 (6)
H24	0.196272	0.548059	0.861430	0.032*
C25	0.1375 (3)	0.6260 (2)	0.90703 (9)	0.0262 (6)
C26	0.0151 (3)	0.5984 (3)	0.90691 (9)	0.0303 (6)
H26	−0.014665	0.554893	0.888515	0.036*
C27	−0.0594 (3)	0.6349 (3)	0.93353 (11)	0.0333 (7)
C28	−0.0190 (3)	0.6997 (3)	0.96125 (10)	0.0369 (8)
H28	−0.073061	0.724205	0.979370	0.044*
C29	0.0990 (3)	0.7270 (3)	0.96179 (10)	0.0342 (8)
H29	0.126805	0.770477	0.980477	0.041*
C30	0.1804 (3)	0.6910 (2)	0.93480 (9)	0.0279 (6)
C31	0.3688 (3)	0.6911 (2)	0.90978 (9)	0.0260 (6)
C32	0.5181 (3)	0.7945 (3)	0.93649 (11)	0.0380 (8)
H32A	0.601622	0.813868	0.932406	0.057*
H32B	0.510293	0.764395	0.961050	0.057*
H32C	0.466450	0.852445	0.935054	0.057*
Br2	1.50319 (4)	0.52837 (3)	0.98077 (2)	0.04694 (11)
O3	1.1137 (2)	0.17437 (18)	0.87033 (7)	0.0340 (5)
H3O	1.149698	0.120544	0.872362	0.051*
O4	0.8318 (2)	0.3046 (2)	0.95890 (8)	0.0422 (6)
N3	1.1594 (3)	−0.0081 (2)	0.89439 (9)	0.0374 (7)
N4	0.9864 (3)	0.3925 (2)	0.98480 (8)	0.0365 (7)
C33	0.9444 (3)	0.2672 (2)	0.89183 (9)	0.0295 (7)
H33	0.859615	0.258280	0.900123	0.035*
C34	0.9919 (3)	0.1632 (2)	0.88238 (9)	0.0291 (6)
C35	0.9948 (3)	0.0969 (2)	0.91712 (9)	0.0321 (7)
H35A	1.050174	0.125785	0.935591	0.039*
H35B	0.913825	0.095086	0.928300	0.039*
C36	1.0347 (3)	−0.0074 (3)	0.90853 (11)	0.0365 (8)
H36A	1.029756	−0.047775	0.931298	0.044*

H36B	0.980637	−0.036456	0.889749	0.044*
C37	1.2456 (4)	−0.0081 (4)	0.92483 (14)	0.0522 (11)
H37A	1.327104	−0.004144	0.914792	0.078*
H37B	1.237028	−0.068711	0.939203	0.078*
H37C	1.230662	0.048388	0.940944	0.078*
C38	1.1821 (4)	−0.0913 (3)	0.86971 (13)	0.0510 (11)
H38A	1.126162	−0.089017	0.848772	0.077*
H38B	1.170719	−0.152677	0.883403	0.077*
H38C	1.264538	−0.087986	0.860431	0.077*
C39	0.9167 (3)	0.1173 (2)	0.85070 (10)	0.0331 (8)
C40	0.9708 (4)	0.1040 (3)	0.81713 (11)	0.0480 (10)
H40	1.050579	0.126813	0.814025	0.058*
C41	0.9141 (5)	0.0580 (4)	0.78685 (12)	0.0572 (13)
H41	0.955036	0.052016	0.763861	0.069*
C42	0.8021 (4)	0.0226 (3)	0.79045 (11)	0.0489 (11)
H42	0.764721	−0.009785	0.770191	0.059*
C43	0.7401 (4)	0.0336 (3)	0.82435 (10)	0.0370 (8)
C44	0.6229 (4)	−0.0050 (3)	0.82814 (12)	0.0438 (9)
H44	0.587964	−0.039705	0.808023	0.053*
C45	0.5592 (4)	0.0068 (3)	0.86000 (13)	0.0468 (10)
H45	0.481437	−0.020876	0.862275	0.056*
C46	0.6092 (4)	0.0603 (3)	0.88956 (11)	0.0389 (8)
H46	0.563583	0.070801	0.911439	0.047*
C47	0.7224 (3)	0.0971 (3)	0.88713 (9)	0.0328 (7)
H47	0.754336	0.132226	0.907619	0.039*
C48	0.7944 (3)	0.0846 (2)	0.85485 (10)	0.0319 (7)
C49	0.9402 (3)	0.3399 (2)	0.85952 (10)	0.0283 (7)
C50	0.8419 (3)	0.4007 (3)	0.85705 (11)	0.0366 (8)
H50	0.777022	0.391304	0.873727	0.044*
C51	0.8348 (4)	0.4754 (3)	0.83090 (12)	0.0438 (9)
H51	0.766337	0.516697	0.830043	0.053*
C52	0.9274 (4)	0.4890 (3)	0.80626 (12)	0.0442 (9)
H52	0.922986	0.539544	0.788192	0.053*
C53	1.0272 (4)	0.4288 (3)	0.80792 (11)	0.0459 (10)
H53	1.091198	0.438060	0.790888	0.055*
C54	1.0343 (3)	0.3547 (3)	0.83440 (11)	0.0376 (8)
H54	1.103315	0.313947	0.835444	0.045*
C55	1.0098 (3)	0.3161 (2)	0.92410 (9)	0.0280 (6)
C56	1.1266 (3)	0.3448 (2)	0.92281 (9)	0.0298 (7)
H56	1.173876	0.328393	0.901803	0.036*
C57	1.1792 (3)	0.3992 (3)	0.95242 (9)	0.0291 (7)
C58	1.2989 (3)	0.4300 (3)	0.95237 (10)	0.0315 (7)
H58	1.350752	0.411765	0.932661	0.038*
C59	1.3406 (3)	0.4866 (3)	0.98087 (11)	0.0353 (7)
C60	1.2659 (4)	0.5160 (3)	1.01026 (10)	0.0410 (9)
H60	1.296029	0.556768	1.029467	0.049*
C61	1.1491 (4)	0.4850 (3)	1.01081 (10)	0.0414 (9)
H61	1.098304	0.504605	1.030619	0.050*

C62	1.1029 (3)	0.4246 (2)	0.98254 (10)	0.0320 (7)
C63	0.9454 (3)	0.3401 (3)	0.95731 (10)	0.0316 (7)
C64	0.7628 (4)	0.3276 (4)	0.99162 (13)	0.0553 (12)
H64A	0.755306	0.398868	0.994016	0.083*
H64B	0.803387	0.301156	1.013550	0.083*
H64C	0.682908	0.298312	0.989498	0.083*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02814 (17)	0.0671 (3)	0.0478 (2)	−0.00323 (18)	0.00642 (17)	−0.0063 (2)
O1	0.0292 (11)	0.0352 (12)	0.0256 (12)	−0.0012 (10)	−0.0035 (9)	−0.0023 (10)
O2	0.0302 (13)	0.0367 (12)	0.0264 (11)	−0.0050 (10)	−0.0010 (10)	−0.0094 (10)
N1	0.0375 (18)	0.0485 (19)	0.0262 (15)	0.0042 (14)	−0.0004 (12)	0.0038 (14)
N2	0.0315 (15)	0.0329 (14)	0.0223 (13)	−0.0007 (11)	0.0021 (11)	−0.0026 (12)
C1	0.0232 (14)	0.0247 (15)	0.0269 (15)	−0.0009 (12)	0.0017 (11)	−0.0013 (13)
C2	0.0264 (15)	0.0260 (16)	0.0258 (16)	−0.0017 (12)	0.0012 (12)	−0.0015 (13)
C3	0.0282 (16)	0.0296 (17)	0.0285 (17)	0.0027 (13)	0.0001 (13)	−0.0026 (13)
C4	0.0321 (18)	0.0327 (18)	0.0344 (19)	0.0013 (14)	0.0026 (14)	0.0046 (15)
C5	0.037 (2)	0.078 (3)	0.050 (3)	0.010 (2)	−0.0086 (19)	0.005 (2)
C6	0.072 (3)	0.062 (3)	0.029 (2)	0.005 (2)	0.002 (2)	0.010 (2)
C7	0.0307 (17)	0.0271 (16)	0.0298 (17)	0.0035 (12)	0.0046 (13)	0.0022 (13)
C8	0.043 (2)	0.0351 (19)	0.0295 (17)	0.0049 (15)	0.0079 (14)	−0.0011 (15)
C9	0.051 (2)	0.042 (2)	0.037 (2)	0.0125 (19)	0.0171 (17)	0.0035 (18)
C10	0.046 (2)	0.052 (2)	0.045 (2)	0.013 (2)	0.023 (2)	0.0110 (19)
C11	0.033 (2)	0.040 (2)	0.056 (3)	0.0050 (15)	0.0146 (17)	0.0145 (19)
C12	0.033 (2)	0.059 (3)	0.086 (4)	−0.001 (2)	0.020 (2)	0.020 (3)
C13	0.033 (2)	0.059 (3)	0.096 (4)	−0.010 (2)	0.007 (2)	0.005 (3)
C14	0.036 (2)	0.052 (2)	0.071 (3)	−0.015 (2)	−0.004 (2)	0.007 (2)
C15	0.0329 (19)	0.0330 (19)	0.047 (2)	−0.0018 (15)	−0.0004 (16)	0.0034 (17)
C16	0.0319 (18)	0.0290 (18)	0.040 (2)	0.0031 (14)	0.0044 (15)	0.0076 (15)
C17	0.0270 (15)	0.0264 (16)	0.0279 (16)	0.0022 (13)	0.0045 (12)	−0.0035 (13)
C18	0.037 (2)	0.0321 (17)	0.039 (2)	0.0034 (14)	0.0005 (15)	0.0015 (15)
C19	0.041 (2)	0.036 (2)	0.059 (3)	0.0074 (16)	0.0006 (18)	0.0106 (19)
C20	0.043 (2)	0.0276 (18)	0.070 (3)	0.0073 (16)	0.014 (2)	0.0029 (19)
C21	0.043 (2)	0.0295 (19)	0.064 (3)	−0.0002 (16)	0.003 (2)	−0.0094 (18)
C22	0.038 (2)	0.0273 (18)	0.047 (2)	−0.0003 (14)	−0.0023 (16)	−0.0066 (16)
C23	0.0311 (16)	0.0241 (15)	0.0213 (15)	0.0004 (12)	0.0019 (12)	−0.0010 (12)
C24	0.0297 (15)	0.0251 (15)	0.0251 (15)	−0.0003 (13)	0.0014 (13)	−0.0024 (13)
C25	0.0264 (15)	0.0256 (16)	0.0265 (16)	0.0001 (12)	0.0001 (12)	0.0025 (12)
C26	0.0284 (16)	0.0324 (16)	0.0301 (16)	0.0025 (14)	0.0030 (13)	−0.0006 (14)
C27	0.0232 (15)	0.0424 (19)	0.0342 (18)	0.0026 (13)	0.0019 (14)	0.0022 (16)
C28	0.0317 (19)	0.049 (2)	0.0299 (18)	0.0033 (15)	0.0058 (14)	−0.0046 (15)
C29	0.0317 (18)	0.044 (2)	0.0270 (17)	0.0024 (15)	0.0028 (14)	−0.0069 (15)
C30	0.0316 (16)	0.0300 (16)	0.0220 (15)	0.0037 (12)	0.0000 (13)	0.0022 (14)
C31	0.0255 (15)	0.0282 (16)	0.0242 (15)	−0.0005 (12)	−0.0035 (12)	0.0007 (13)
C32	0.0321 (18)	0.047 (2)	0.0349 (18)	−0.0078 (15)	−0.0054 (15)	−0.0130 (16)
Br2	0.0435 (2)	0.0550 (2)	0.0423 (2)	−0.00769 (19)	−0.00983 (19)	−0.00133 (19)

O3	0.0301 (12)	0.0305 (12)	0.0415 (14)	0.0025 (10)	0.0129 (11)	−0.0005 (11)
O4	0.0351 (14)	0.0499 (16)	0.0415 (16)	−0.0055 (12)	0.0175 (12)	−0.0080 (13)
N3	0.0394 (17)	0.0342 (17)	0.0387 (17)	0.0073 (13)	0.0039 (14)	−0.0059 (13)
N4	0.0492 (18)	0.0350 (15)	0.0253 (14)	0.0036 (14)	0.0091 (13)	0.0005 (12)
C33	0.0293 (16)	0.0318 (17)	0.0274 (17)	0.0002 (13)	0.0044 (13)	−0.0012 (13)
C34	0.0295 (16)	0.0280 (15)	0.0298 (16)	−0.0017 (13)	0.0078 (14)	−0.0003 (12)
C35	0.0337 (16)	0.0310 (16)	0.0316 (16)	0.0022 (15)	0.0063 (14)	0.0028 (13)
C36	0.0364 (19)	0.0334 (18)	0.040 (2)	0.0002 (14)	0.0039 (15)	0.0014 (15)
C37	0.040 (2)	0.058 (3)	0.059 (3)	0.0083 (18)	−0.0011 (19)	−0.009 (2)
C38	0.066 (3)	0.037 (2)	0.049 (2)	0.011 (2)	0.011 (2)	−0.0114 (19)
C39	0.043 (2)	0.0280 (18)	0.0279 (17)	−0.0008 (14)	0.0087 (14)	0.0006 (14)
C40	0.054 (3)	0.055 (2)	0.036 (2)	−0.013 (2)	0.0160 (18)	−0.0104 (19)
C41	0.075 (3)	0.066 (3)	0.031 (2)	−0.017 (2)	0.013 (2)	−0.014 (2)
C42	0.069 (3)	0.047 (2)	0.031 (2)	−0.009 (2)	0.0019 (18)	−0.0097 (18)
C43	0.047 (2)	0.0289 (17)	0.0348 (18)	−0.0035 (16)	−0.0022 (15)	−0.0009 (15)
C44	0.048 (2)	0.040 (2)	0.044 (2)	−0.0053 (17)	−0.0108 (18)	−0.0002 (17)
C45	0.045 (2)	0.042 (2)	0.053 (3)	−0.0093 (17)	−0.009 (2)	0.0095 (19)
C46	0.0363 (19)	0.042 (2)	0.039 (2)	−0.0012 (16)	0.0024 (16)	0.0106 (16)
C47	0.0379 (17)	0.0302 (16)	0.0302 (17)	−0.0001 (16)	0.0023 (14)	0.0024 (14)
C48	0.041 (2)	0.0246 (16)	0.0305 (17)	−0.0002 (14)	0.0021 (14)	0.0027 (13)
C49	0.0282 (16)	0.0276 (16)	0.0290 (17)	−0.0015 (13)	0.0010 (13)	−0.0028 (13)
C50	0.0339 (18)	0.0365 (19)	0.039 (2)	0.0036 (16)	0.0004 (15)	−0.0022 (17)
C51	0.045 (2)	0.041 (2)	0.044 (2)	0.0093 (18)	−0.0049 (17)	0.0024 (19)
C52	0.051 (2)	0.043 (2)	0.039 (2)	0.0023 (18)	−0.0076 (18)	0.0110 (17)
C53	0.041 (2)	0.058 (3)	0.039 (2)	0.0013 (18)	0.0032 (16)	0.0168 (18)
C54	0.0289 (18)	0.047 (2)	0.037 (2)	0.0062 (15)	0.0027 (14)	0.0085 (16)
C55	0.0308 (16)	0.0248 (14)	0.0283 (16)	0.0043 (13)	0.0044 (13)	0.0014 (12)
C56	0.0375 (18)	0.0282 (17)	0.0236 (16)	−0.0006 (13)	0.0062 (13)	−0.0008 (13)
C57	0.0353 (17)	0.0286 (16)	0.0234 (15)	0.0026 (14)	0.0032 (13)	0.0040 (14)
C58	0.0327 (18)	0.0343 (18)	0.0275 (17)	0.0014 (13)	0.0011 (13)	0.0023 (14)
C59	0.0399 (19)	0.0352 (18)	0.0309 (17)	−0.0005 (14)	−0.0068 (15)	0.0049 (16)
C60	0.055 (2)	0.042 (2)	0.0260 (17)	0.0029 (19)	−0.0049 (16)	−0.0027 (15)
C61	0.061 (3)	0.039 (2)	0.0234 (17)	0.0043 (18)	0.0039 (16)	−0.0042 (15)
C62	0.0422 (19)	0.0289 (16)	0.0250 (16)	0.0041 (14)	0.0035 (15)	0.0022 (14)
C63	0.0336 (17)	0.0299 (17)	0.0312 (18)	0.0020 (14)	0.0080 (14)	0.0022 (14)
C64	0.047 (3)	0.070 (3)	0.049 (3)	−0.002 (2)	0.030 (2)	−0.004 (2)

Geometric parameters (Å, º)

Br1—C27	1.903 (3)	Br2—C59	1.902 (4)
O1—C2	1.421 (4)	O3—C34	1.435 (4)
O1—H1O	0.8400	O3—H3O	0.8400
O2—C31	1.354 (4)	O4—C63	1.358 (4)
O2—C32	1.437 (4)	O4—C64	1.443 (4)
N1—C5	1.461 (5)	N3—C37	1.460 (6)
N1—C4	1.470 (5)	N3—C38	1.464 (5)
N1—C6	1.477 (5)	N3—C36	1.482 (5)
N2—C31	1.311 (4)	N4—C63	1.305 (5)

N2—C30	1.372 (4)	N4—C62	1.374 (5)
C1—C17	1.526 (4)	C33—C55	1.526 (5)
C1—C23	1.531 (4)	C33—C49	1.531 (5)
C1—C2	1.560 (5)	C33—C34	1.552 (5)
C1—H1	1.0000	C33—H33	1.0000
C2—C3	1.545 (5)	C34—C35	1.546 (5)
C2—C7	1.550 (5)	C34—C39	1.550 (5)
C3—C4	1.530 (5)	C35—C36	1.524 (5)
C3—H3A	0.9900	C35—H35A	0.9900
C3—H3B	0.9900	C35—H35B	0.9900
C4—H4A	0.9900	C36—H36A	0.9900
C4—H4B	0.9900	C36—H36B	0.9900
C5—H5A	0.9800	C37—H37A	0.9800
C5—H5B	0.9800	C37—H37B	0.9800
C5—H5C	0.9800	C37—H37C	0.9800
C6—H6A	0.9800	C38—H38A	0.9800
C6—H6B	0.9800	C38—H38B	0.9800
C6—H6C	0.9800	C38—H38C	0.9800
C7—C8	1.377 (5)	C39—C40	1.365 (5)
C7—C16	1.449 (5)	C39—C48	1.443 (5)
C8—C9	1.417 (5)	C40—C41	1.409 (6)
C8—H8	0.9500	C40—H40	0.9500
C9—C10	1.346 (7)	C41—C42	1.347 (7)
C9—H9	0.9500	C41—H41	0.9500
C10—C11	1.413 (6)	C42—C43	1.412 (6)
C10—H10	0.9500	C42—H42	0.9500
C11—C12	1.404 (7)	C43—C44	1.416 (6)
C11—C16	1.438 (5)	C43—C48	1.436 (5)
C12—C13	1.366 (8)	C44—C45	1.360 (6)
C12—H12	0.9500	C44—H44	0.9500
C13—C14	1.384 (7)	C45—C46	1.407 (6)
C13—H13	0.9500	C45—H45	0.9500
C14—C15	1.371 (6)	C46—C47	1.362 (5)
C14—H14	0.9500	C46—H46	0.9500
C15—C16	1.420 (6)	C47—C48	1.425 (5)
C15—H15	0.9500	C47—H47	0.9500
C17—C22	1.391 (5)	C49—C50	1.378 (5)
C17—C18	1.395 (5)	C49—C54	1.402 (5)
C18—C19	1.388 (5)	C50—C51	1.390 (6)
C18—H18	0.9500	C50—H50	0.9500
C19—C20	1.382 (6)	C51—C52	1.376 (6)
C19—H19	0.9500	C51—H51	0.9500
C20—C21	1.372 (6)	C52—C53	1.385 (6)
C20—H20	0.9500	C52—H52	0.9500
C21—C22	1.385 (5)	C53—C54	1.393 (5)
C21—H21	0.9500	C53—H53	0.9500
C22—H22	0.9500	C54—H54	0.9500
C23—C24	1.361 (5)	C55—C56	1.362 (5)

C23—C31	1.435 (4)	C55—C63	1.435 (5)
C24—C25	1.424 (4)	C56—C57	1.426 (5)
C24—H24	0.9500	C56—H56	0.9500
C25—C26	1.416 (5)	C57—C58	1.400 (5)
C25—C30	1.421 (5)	C57—C62	1.423 (5)
C26—C27	1.365 (5)	C58—C59	1.367 (5)
C26—H26	0.9500	C58—H58	0.9500
C27—C28	1.409 (5)	C59—C60	1.406 (6)
C28—C29	1.369 (5)	C60—C61	1.371 (6)
C28—H28	0.9500	C60—H60	0.9500
C29—C30	1.418 (5)	C61—C62	1.409 (5)
C29—H29	0.9500	C61—H61	0.9500
C32—H32A	0.9800	C64—H64A	0.9800
C32—H32B	0.9800	C64—H64B	0.9800
C32—H32C	0.9800	C64—H64C	0.9800
C2—O1—H1O	109.5	C34—O3—H3O	109.5
C31—O2—C32	117.4 (3)	C63—O4—C64	117.1 (3)
C5—N1—C4	111.0 (3)	C37—N3—C38	110.1 (3)
C5—N1—C6	110.1 (4)	C37—N3—C36	111.1 (3)
C4—N1—C6	111.0 (3)	C38—N3—C36	112.1 (3)
C31—N2—C30	117.1 (3)	C63—N4—C62	117.5 (3)
C17—C1—C23	109.9 (3)	C55—C33—C49	108.2 (3)
C17—C1—C2	116.8 (3)	C55—C33—C34	113.8 (3)
C23—C1—C2	112.2 (3)	C49—C33—C34	115.8 (3)
C17—C1—H1	105.7	C55—C33—H33	106.1
C23—C1—H1	105.7	C49—C33—H33	106.1
C2—C1—H1	105.7	C34—C33—H33	106.1
O1—C2—C3	106.7 (3)	O3—C34—C35	106.7 (3)
O1—C2—C7	110.2 (3)	O3—C34—C39	109.5 (3)
C3—C2—C7	109.0 (3)	C35—C34—C39	111.8 (3)
O1—C2—C1	107.1 (3)	O3—C34—C33	107.0 (3)
C3—C2—C1	112.1 (3)	C35—C34—C33	111.3 (3)
C7—C2—C1	111.7 (3)	C39—C34—C33	110.3 (3)
C4—C3—C2	112.8 (3)	C36—C35—C34	112.8 (3)
C4—C3—H3A	109.0	C36—C35—H35A	109.0
C2—C3—H3A	109.0	C34—C35—H35A	109.0
C4—C3—H3B	109.0	C36—C35—H35B	109.0
C2—C3—H3B	109.0	C34—C35—H35B	109.0
H3A—C3—H3B	107.8	H35A—C35—H35B	107.8
N1—C4—C3	112.2 (3)	N3—C36—C35	110.5 (3)
N1—C4—H4A	109.2	N3—C36—H36A	109.5
C3—C4—H4A	109.2	C35—C36—H36A	109.5
N1—C4—H4B	109.2	N3—C36—H36B	109.5
C3—C4—H4B	109.2	C35—C36—H36B	109.5
H4A—C4—H4B	107.9	H36A—C36—H36B	108.1
N1—C5—H5A	109.5	N3—C37—H37A	109.5
N1—C5—H5B	109.5	N3—C37—H37B	109.5

H5A—C5—H5B	109.5	H37A—C37—H37B	109.5
N1—C5—H5C	109.5	N3—C37—H37C	109.5
H5A—C5—H5C	109.5	H37A—C37—H37C	109.5
H5B—C5—H5C	109.5	H37B—C37—H37C	109.5
N1—C6—H6A	109.5	N3—C38—H38A	109.5
N1—C6—H6B	109.5	N3—C38—H38B	109.5
H6A—C6—H6B	109.5	H38A—C38—H38B	109.5
N1—C6—H6C	109.5	N3—C38—H38C	109.5
H6A—C6—H6C	109.5	H38A—C38—H38C	109.5
H6B—C6—H6C	109.5	H38B—C38—H38C	109.5
C8—C7—C16	118.4 (3)	C40—C39—C48	118.0 (4)
C8—C7—C2	118.0 (3)	C40—C39—C34	117.9 (3)
C16—C7—C2	123.2 (3)	C48—C39—C34	124.1 (3)
C7—C8—C9	122.2 (4)	C39—C40—C41	123.2 (4)
C7—C8—H8	118.9	C39—C40—H40	118.4
C9—C8—H8	118.9	C41—C40—H40	118.4
C10—C9—C8	120.2 (4)	C42—C41—C40	120.1 (4)
C10—C9—H9	119.9	C42—C41—H41	119.9
C8—C9—H9	119.9	C40—C41—H41	119.9
C9—C10—C11	120.9 (4)	C41—C42—C43	120.0 (4)
C9—C10—H10	119.5	C41—C42—H42	120.0
C11—C10—H10	119.5	C43—C42—H42	120.0
C12—C11—C10	120.4 (4)	C42—C43—C44	119.8 (4)
C12—C11—C16	119.6 (4)	C42—C43—C48	120.5 (4)
C10—C11—C16	119.9 (4)	C44—C43—C48	119.7 (4)
C13—C12—C11	121.3 (4)	C45—C44—C43	121.3 (4)
C13—C12—H12	119.4	C45—C44—H44	119.3
C11—C12—H12	119.4	C43—C44—H44	119.3
C12—C13—C14	120.0 (4)	C44—C45—C46	119.6 (4)
C12—C13—H13	120.0	C44—C45—H45	120.2
C14—C13—H13	120.0	C46—C45—H45	120.2
C15—C14—C13	120.7 (5)	C47—C46—C45	120.7 (4)
C15—C14—H14	119.7	C47—C46—H46	119.7
C13—C14—H14	119.7	C45—C46—H46	119.7
C14—C15—C16	121.8 (4)	C46—C47—C48	122.1 (4)
C14—C15—H15	119.1	C46—C47—H47	119.0
C16—C15—H15	119.1	C48—C47—H47	119.0
C15—C16—C11	116.5 (4)	C47—C48—C43	116.5 (3)
C15—C16—C7	125.3 (3)	C47—C48—C39	125.5 (3)
C11—C16—C7	118.2 (4)	C43—C48—C39	118.0 (3)
C22—C17—C18	117.3 (3)	C50—C49—C54	117.9 (3)
C22—C17—C1	125.2 (3)	C50—C49—C33	117.6 (3)
C18—C17—C1	117.3 (3)	C54—C49—C33	124.2 (3)
C19—C18—C17	121.3 (4)	C49—C50—C51	122.0 (4)
C19—C18—H18	119.4	C49—C50—H50	119.0
C17—C18—H18	119.4	C51—C50—H50	119.0
C20—C19—C18	120.5 (4)	C52—C51—C50	119.6 (4)
C20—C19—H19	119.7	C52—C51—H51	120.2

C18—C19—H19	119.7	C50—C51—H51	120.2
C21—C20—C19	118.5 (4)	C51—C52—C53	119.7 (4)
C21—C20—H20	120.7	C51—C52—H52	120.1
C19—C20—H20	120.7	C53—C52—H52	120.1
C20—C21—C22	121.4 (4)	C52—C53—C54	120.4 (4)
C20—C21—H21	119.3	C52—C53—H53	119.8
C22—C21—H21	119.3	C54—C53—H53	119.8
C21—C22—C17	120.9 (4)	C53—C54—C49	120.3 (3)
C21—C22—H22	119.5	C53—C54—H54	119.8
C17—C22—H22	119.5	C49—C54—H54	119.8
C24—C23—C31	116.5 (3)	C56—C55—C63	116.2 (3)
C24—C23—C1	124.3 (3)	C56—C55—C33	123.8 (3)
C31—C23—C1	119.2 (3)	C63—C55—C33	119.8 (3)
C23—C24—C25	120.8 (3)	C55—C56—C57	121.2 (3)
C23—C24—H24	119.6	C55—C56—H56	119.4
C25—C24—H24	119.6	C57—C56—H56	119.4
C26—C25—C30	119.6 (3)	C58—C57—C62	119.9 (3)
C26—C25—C24	123.1 (3)	C58—C57—C56	123.2 (3)
C30—C25—C24	117.3 (3)	C62—C57—C56	116.9 (3)
C27—C26—C25	119.2 (3)	C59—C58—C57	119.6 (3)
C27—C26—H26	120.4	C59—C58—H58	120.2
C25—C26—H26	120.4	C57—C58—H58	120.2
C26—C27—C28	122.2 (3)	C58—C59—C60	121.7 (4)
C26—C27—Br1	120.1 (3)	C58—C59—Br2	119.5 (3)
C28—C27—Br1	117.7 (3)	C60—C59—Br2	118.8 (3)
C29—C28—C27	119.3 (3)	C61—C60—C59	119.1 (4)
C29—C28—H28	120.4	C61—C60—H60	120.4
C27—C28—H28	120.4	C59—C60—H60	120.4
C28—C29—C30	120.8 (3)	C60—C61—C62	121.2 (4)
C28—C29—H29	119.6	C60—C61—H61	119.4
C30—C29—H29	119.6	C62—C61—H61	119.4
N2—C30—C29	118.5 (3)	N4—C62—C61	119.3 (3)
N2—C30—C25	122.5 (3)	N4—C62—C57	122.3 (3)
C29—C30—C25	118.9 (3)	C61—C62—C57	118.4 (4)
N2—C31—O2	119.0 (3)	N4—C63—O4	119.4 (3)
N2—C31—C23	125.8 (3)	N4—C63—C55	125.7 (3)
O2—C31—C23	115.2 (3)	O4—C63—C55	114.9 (3)
O2—C32—H32A	109.5	O4—C64—H64A	109.5
O2—C32—H32B	109.5	O4—C64—H64B	109.5
H32A—C32—H32B	109.5	H64A—C64—H64B	109.5
O2—C32—H32C	109.5	O4—C64—H64C	109.5
H32A—C32—H32C	109.5	H64A—C64—H64C	109.5
H32B—C32—H32C	109.5	H64B—C64—H64C	109.5
C17—C1—C2—O1	52.3 (4)	C55—C33—C34—O3	−63.8 (3)
C23—C1—C2—O1	−75.9 (3)	C49—C33—C34—O3	62.5 (4)
C17—C1—C2—C3	169.0 (3)	C55—C33—C34—C35	52.5 (4)
C23—C1—C2—C3	40.8 (4)	C49—C33—C34—C35	178.8 (3)

C17—C1—C2—C7	−68.4 (4)	C55—C33—C34—C39	177.2 (3)
C23—C1—C2—C7	163.4 (3)	C49—C33—C34—C39	−56.5 (4)
O1—C2—C3—C4	−67.4 (3)	O3—C34—C35—C36	−66.6 (4)
C7—C2—C3—C4	51.6 (4)	C39—C34—C35—C36	53.1 (4)
C1—C2—C3—C4	175.7 (3)	C33—C34—C35—C36	177.0 (3)
C5—N1—C4—C3	83.1 (4)	C37—N3—C36—C35	84.3 (4)
C6—N1—C4—C3	−154.1 (3)	C38—N3—C36—C35	−152.0 (3)
C2—C3—C4—N1	58.7 (4)	C34—C35—C36—N3	63.0 (4)
O1—C2—C7—C8	−0.5 (4)	O3—C34—C39—C40	−5.9 (5)
C3—C2—C7—C8	−117.3 (3)	C35—C34—C39—C40	−123.9 (4)
C1—C2—C7—C8	118.3 (3)	C33—C34—C39—C40	111.6 (4)
O1—C2—C7—C16	171.3 (3)	O3—C34—C39—C48	171.9 (3)
C3—C2—C7—C16	54.5 (4)	C35—C34—C39—C48	53.8 (4)
C1—C2—C7—C16	−69.8 (4)	C33—C34—C39—C48	−70.7 (4)
C16—C7—C8—C9	−2.8 (5)	C48—C39—C40—C41	−1.6 (7)
C2—C7—C8—C9	169.4 (3)	C34—C39—C40—C41	176.3 (4)
C7—C8—C9—C10	−0.8 (6)	C39—C40—C41—C42	−1.6 (8)
C8—C9—C10—C11	2.5 (6)	C40—C41—C42—C43	1.5 (8)
C9—C10—C11—C12	178.7 (4)	C41—C42—C43—C44	−179.2 (5)
C9—C10—C11—C16	−0.6 (6)	C41—C42—C43—C48	1.6 (7)
C10—C11—C12—C13	−176.8 (5)	C42—C43—C44—C45	−178.0 (4)
C16—C11—C12—C13	2.4 (7)	C48—C43—C44—C45	1.2 (6)
C11—C12—C13—C14	−0.7 (8)	C43—C44—C45—C46	1.5 (6)
C12—C13—C14—C15	−1.2 (8)	C44—C45—C46—C47	−2.5 (6)
C13—C14—C15—C16	1.5 (7)	C45—C46—C47—C48	0.8 (6)
C14—C15—C16—C11	0.2 (6)	C46—C47—C48—C43	1.9 (5)
C14—C15—C16—C7	−179.7 (4)	C46—C47—C48—C39	−177.0 (3)
C12—C11—C16—C15	−2.1 (6)	C42—C43—C48—C47	176.4 (4)
C10—C11—C16—C15	177.2 (4)	C44—C43—C48—C47	−2.8 (5)
C12—C11—C16—C7	177.8 (4)	C42—C43—C48—C39	−4.7 (5)
C10—C11—C16—C7	−2.9 (5)	C44—C43—C48—C39	176.2 (3)
C8—C7—C16—C15	−175.6 (4)	C40—C39—C48—C47	−176.6 (4)
C2—C7—C16—C15	12.6 (6)	C34—C39—C48—C47	5.7 (5)
C8—C7—C16—C11	4.6 (5)	C40—C39—C48—C43	4.5 (5)
C2—C7—C16—C11	−167.3 (3)	C34—C39—C48—C43	−173.2 (3)
C23—C1—C17—C22	81.5 (4)	C55—C33—C49—C50	−91.5 (4)
C2—C1—C17—C22	−47.9 (5)	C34—C33—C49—C50	139.4 (3)
C23—C1—C17—C18	−93.4 (4)	C55—C33—C49—C54	82.5 (4)
C2—C1—C17—C18	137.3 (3)	C34—C33—C49—C54	−46.6 (5)
C22—C17—C18—C19	−1.0 (6)	C54—C49—C50—C51	−0.6 (6)
C1—C17—C18—C19	174.2 (4)	C33—C49—C50—C51	173.8 (4)
C17—C18—C19—C20	0.5 (6)	C49—C50—C51—C52	0.8 (6)
C18—C19—C20—C21	0.1 (7)	C50—C51—C52—C53	−0.5 (7)
C19—C20—C21—C22	−0.1 (7)	C51—C52—C53—C54	−0.1 (7)
C20—C21—C22—C17	−0.4 (7)	C52—C53—C54—C49	0.4 (6)
C18—C17—C22—C21	0.9 (6)	C50—C49—C54—C53	0.0 (6)
C1—C17—C22—C21	−173.9 (4)	C33—C49—C54—C53	−174.0 (4)
C17—C1—C23—C24	−54.5 (4)	C49—C33—C55—C56	−63.1 (4)

C2—C1—C23—C24	77.3 (4)	C34—C33—C55—C56	67.1 (4)
C17—C1—C23—C31	126.0 (3)	C49—C33—C55—C63	112.6 (3)
C2—C1—C23—C31	−102.2 (3)	C34—C33—C55—C63	−117.2 (3)
C31—C23—C24—C25	−0.6 (5)	C63—C55—C56—C57	−2.4 (5)
C1—C23—C24—C25	179.8 (3)	C33—C55—C56—C57	173.4 (3)
C23—C24—C25—C26	178.3 (3)	C55—C56—C57—C58	179.8 (3)
C23—C24—C25—C30	−1.1 (5)	C55—C56—C57—C62	−2.1 (5)
C30—C25—C26—C27	0.1 (5)	C62—C57—C58—C59	−1.7 (5)
C24—C25—C26—C27	−179.3 (3)	C56—C57—C58—C59	176.3 (3)
C25—C26—C27—C28	0.1 (5)	C57—C58—C59—C60	−0.8 (5)
C25—C26—C27—Br1	−180.0 (3)	C57—C58—C59—Br2	180.0 (3)
C26—C27—C28—C29	−0.3 (6)	C58—C59—C60—C61	1.7 (6)
Br1—C27—C28—C29	179.8 (3)	Br2—C59—C60—C61	−179.1 (3)
C27—C28—C29—C30	0.2 (6)	C59—C60—C61—C62	−0.1 (6)
C31—N2—C30—C29	−177.9 (3)	C63—N4—C62—C61	177.2 (3)
C31—N2—C30—C25	−0.3 (5)	C63—N4—C62—C57	−2.6 (5)
C28—C29—C30—N2	177.8 (3)	C60—C61—C62—N4	177.9 (3)
C28—C29—C30—C25	0.0 (5)	C60—C61—C62—C57	−2.4 (5)
C26—C25—C30—N2	−177.8 (3)	C58—C57—C62—N4	−177.0 (3)
C24—C25—C30—N2	1.6 (5)	C56—C57—C62—N4	4.8 (5)
C26—C25—C30—C29	−0.2 (5)	C58—C57—C62—C61	3.3 (5)
C24—C25—C30—C29	179.3 (3)	C56—C57—C62—C61	−174.9 (3)
C30—N2—C31—O2	176.5 (3)	C62—N4—C63—O4	178.1 (3)
C30—N2—C31—C23	−1.7 (5)	C62—N4—C63—C55	−2.6 (5)
C32—O2—C31—N2	0.6 (5)	C64—O4—C63—N4	−0.1 (5)
C32—O2—C31—C23	179.0 (3)	C64—O4—C63—C55	−179.4 (3)
C24—C23—C31—N2	2.1 (5)	C56—C55—C63—N4	5.1 (5)
C1—C23—C31—N2	−178.3 (3)	C33—C55—C63—N4	−170.9 (3)
C24—C23—C31—O2	−176.1 (3)	C56—C55—C63—O4	−175.6 (3)
C1—C23—C31—O2	3.5 (4)	C33—C55—C63—O4	8.4 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···N1	0.84	1.94	2.696 (4)	150
C1—H1···O2	1.00	2.24	2.763 (4)	111
O3—H3*O*···N3	0.84	1.93	2.685 (4)	149
C33—H33···O4	1.00	2.23	2.773 (4)	112

[4-(6-Bromo-2-methoxyquinolin-3-yl)-3-hydroxy-3-(naphthalen-1-yl)-4-phenylbutyl]dimethylazanium 3-carboxyprop-2-enoate (2)

Crystal data

$C_{32}H_{32}BrN_2O_2^+·C_4H_3O_4^-$
M_r = 671.57
Monoclinic, $P2_1$
a = 16.4556 (6) Å
b = 10.3205 (3) Å
c = 20.1636 (8) Å
β = 109.1832 (15)°
V = 3234.2 (2) Å^3
Z = 4
$F(000)$ = 1392

D_x = 1.379 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9891 reflections
θ = 2.9–31.4°

μ = 1.32 mm^{-1}
T = 150 K
Block, colourless
0.45 × 0.37 × 0.17 mm

Data collection

Bruker D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD)
Radiation source: fine focus sealed tube X-ray source
Triumph curved graphite crystal monochromator
Detector resolution: 7.4074 pixels mm^{-1}
ω and phi scans

Absorption correction: multi-scan (SADABS2016; Krause *et al.*, 2015)
T_{min} = 0.438, T_{max} = 0.495
115858 measured reflections
24622 independent reflections
18572 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 33.2°, θ_{min} = 2.3°
h = −25→25
k = −15→15
l = −28→31

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.117
S = 1.06
24622 reflections
837 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 0.4737P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.23 e Å^{-3}
$\Delta\rho_{min}$ = −1.28 e Å^{-3}
Absolute structure: Flack x determined using 7327 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: −0.0144 (14)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1A	0.58678 (3)	0.47935 (4)	0.22528 (2)	0.06533 (14)
O1A	0.82483 (12)	−0.01085 (19)	0.51792 (10)	0.0278 (3)
H1AB	0.828 (2)	0.039 (4)	0.485 (2)	0.042*
O2A	0.77472 (16)	0.3652 (3)	0.64605 (13)	0.0484 (6)
O3A	0.93472 (13)	0.2628 (2)	0.42487 (10)	0.0344 (4)
O4A	0.84664 (14)	0.09248 (19)	0.40198 (9)	0.0349 (4)
O5A	0.81194 (14)	0.52568 (19)	0.20888 (11)	0.0350 (4)
H5A	0.763 (3)	0.555 (4)	0.164 (2)	0.052*
O6A	0.76176 (14)	0.3298 (2)	0.16731 (10)	0.0373 (4)
N1A	1.03839 (12)	0.1521 (2)	0.53959 (11)	0.0244 (4)
H1AN	0.993 (2)	0.179 (4)	0.4961 (19)	0.029*
N2A	0.73312 (15)	0.4780 (3)	0.54157 (16)	0.0405 (6)

C1A	0.75797 (15)	0.1203 (3)	0.58725 (12)	0.0253 (4)
H1A	0.771592	0.149992	0.636934	0.030*
C2A	0.84239 (15)	0.0560 (2)	0.58297 (13)	0.0239 (4)
C3A	0.91264 (15)	0.1580 (2)	0.58576 (14)	0.0262 (4)
H3AA	0.892408	0.215552	0.544294	0.031*
H3AB	0.923897	0.211845	0.628457	0.031*
C4A	0.99581 (16)	0.0896 (3)	0.58663 (15)	0.0314 (5)
H4AA	1.036546	0.089161	0.635301	0.038*
H4AB	0.982420	−0.001652	0.571907	0.038*
C5A	1.0918 (2)	0.2647 (3)	0.57295 (16)	0.0404 (7)
H5AA	1.115756	0.304971	0.539441	0.061*
H5AB	1.138756	0.235812	0.614397	0.061*
H5AC	1.056186	0.327915	0.587129	0.061*
C6A	1.0901 (2)	0.0563 (3)	0.5157 (2)	0.0441 (7)
H6AA	1.051580	−0.006000	0.483812	0.066*
H6AB	1.128727	0.010308	0.556399	0.066*
H6AC	1.124180	0.101322	0.491018	0.066*
C7A	0.87611 (15)	−0.0436 (3)	0.64224 (14)	0.0283 (5)
C8A	0.8828 (2)	−0.1710 (3)	0.62370 (17)	0.0364 (6)
H8A	0.864922	−0.192956	0.575279	0.044*
C9A	0.9150 (2)	−0.2694 (3)	0.6737 (2)	0.0479 (8)
H9A	0.918640	−0.355840	0.658637	0.058*
C10A	0.9408 (2)	−0.2416 (4)	0.7432 (2)	0.0483 (8)
H10A	0.961810	−0.308627	0.776728	0.058*
C11A	0.93654 (19)	−0.1126 (3)	0.76586 (16)	0.0407 (7)
C12A	0.9655 (2)	−0.0838 (5)	0.83857 (17)	0.0526 (9)
H12A	0.986902	−0.151708	0.871500	0.063*
C13A	0.9633 (3)	0.0384 (5)	0.86202 (19)	0.0597 (11)
H13A	0.983221	0.055843	0.911000	0.072*
C14A	0.9313 (2)	0.1395 (4)	0.81378 (16)	0.0479 (8)
H14A	0.929246	0.225084	0.830529	0.058*
C15A	0.90318 (19)	0.1164 (3)	0.74297 (15)	0.0357 (6)
H15A	0.882490	0.186537	0.711397	0.043*
C16A	0.90432 (16)	−0.0111 (3)	0.71559 (14)	0.0316 (5)
C17A	0.67859 (15)	0.0327 (3)	0.57224 (13)	0.0277 (5)
C18A	0.6128 (2)	0.0787 (4)	0.5946 (2)	0.0497 (8)
H18A	0.620591	0.157373	0.620373	0.060*
C19A	0.5355 (2)	0.0117 (4)	0.5799 (2)	0.0569 (10)
H19A	0.490525	0.046866	0.594292	0.068*
C20A	0.52354 (19)	−0.1032 (4)	0.54536 (18)	0.0475 (8)
H20A	0.470901	−0.149129	0.535924	0.057*
C21A	0.5879 (2)	−0.1517 (4)	0.52446 (18)	0.0487 (8)
H21A	0.580561	−0.232974	0.501099	0.058*
C22A	0.66510 (19)	−0.0831 (4)	0.53697 (17)	0.0461 (8)
H22A	0.708730	−0.117411	0.520735	0.055*
C23A	0.73471 (15)	0.2414 (2)	0.54251 (14)	0.0272 (5)
C24A	0.70146 (15)	0.2396 (2)	0.47090 (14)	0.0264 (5)
H24A	0.689599	0.159083	0.446837	0.032*

C25A	0.68451 (16)	0.3567 (3)	0.43217 (16)	0.0307 (5)
C26A	0.64932 (18)	0.3588 (3)	0.35808 (16)	0.0360 (6)
H26A	0.635255	0.280285	0.332208	0.043*
C27A	0.6359 (2)	0.4759 (3)	0.32424 (19)	0.0442 (7)
C28A	0.6564 (2)	0.5939 (3)	0.3598 (2)	0.0473 (8)
H28A	0.648076	0.673357	0.334619	0.057*
C29A	0.68871 (19)	0.5927 (3)	0.4315 (2)	0.0455 (8)
H29A	0.702150	0.672378	0.456212	0.055*
C30A	0.70257 (16)	0.4748 (3)	0.46960 (17)	0.0348 (6)
C31A	0.74691 (17)	0.3681 (3)	0.57451 (17)	0.0361 (6)
C32A	0.7682 (3)	0.4825 (5)	0.6821 (2)	0.0641 (12)
H32A	0.800334	0.551543	0.668372	0.096*
H32B	0.792178	0.468439	0.732896	0.096*
H32C	0.707587	0.507649	0.669651	0.096*
C33A	0.87369 (16)	0.1975 (3)	0.38480 (12)	0.0264 (4)
C34A	0.82928 (16)	0.2472 (3)	0.31203 (13)	0.0267 (5)
H34A	0.779369	0.202920	0.283576	0.032*
C35A	0.85588 (16)	0.3491 (3)	0.28539 (13)	0.0269 (5)
H35A	0.907626	0.391592	0.311789	0.032*
C36A	0.80515 (16)	0.3986 (2)	0.21389 (13)	0.0280 (5)
Br1B	0.03711 (2)	0.42464 (3)	−0.28802 (2)	0.04720 (9)
O1B	0.33662 (11)	0.00508 (18)	0.02269 (9)	0.0267 (3)
H1B	0.327 (2)	0.060 (4)	−0.013 (2)	0.040*
O2B	0.27505 (14)	0.3816 (2)	0.13215 (11)	0.0392 (5)
O3B	0.57136 (13)	0.7488 (3)	0.07837 (11)	0.0460 (6)
O4B	0.68777 (12)	0.62216 (19)	0.10440 (9)	0.0294 (4)
O5B	0.64680 (14)	1.0698 (2)	0.27053 (14)	0.0453 (5)
O6B	0.75267 (14)	0.9246 (2)	0.31105 (10)	0.0398 (5)
H6B	0.771 (3)	0.989 (5)	0.339 (2)	0.060*
N1B	0.53663 (13)	0.1951 (2)	0.04729 (12)	0.0283 (4)
H1BN	0.502 (2)	0.191 (4)	0.006 (2)	0.034*
N2B	0.22630 (13)	0.4745 (2)	0.02299 (12)	0.0303 (4)
C1B	0.25543 (14)	0.1260 (2)	0.08575 (12)	0.0230 (4)
H1BA	0.264502	0.161026	0.133894	0.028*
C2B	0.34548 (14)	0.0737 (2)	0.08604 (12)	0.0229 (4)
C3B	0.40925 (15)	0.1858 (3)	0.08942 (14)	0.0267 (5)
H3BA	0.387922	0.239023	0.046308	0.032*
H3BB	0.413490	0.242027	0.130194	0.032*
C4B	0.49810 (15)	0.1312 (3)	0.09628 (14)	0.0294 (5)
H4BA	0.536885	0.143220	0.145135	0.035*
H4BB	0.493009	0.036984	0.086459	0.035*
C5B	0.55983 (19)	0.3328 (3)	0.06465 (17)	0.0405 (6)
H5BA	0.576770	0.372420	0.026984	0.061*
H5BB	0.607907	0.337836	0.108886	0.061*
H5BC	0.510149	0.379233	0.069535	0.061*
C6B	0.61290 (18)	0.1223 (4)	0.04453 (18)	0.0426 (7)
H6BA	0.597246	0.031375	0.033235	0.064*
H6BB	0.658496	0.127597	0.090215	0.064*

H6BC	0.633488	0.159756	0.008298	0.064*
C7B	0.38230 (15)	−0.0216 (3)	0.14727 (13)	0.0262 (4)
C8B	0.39567 (17)	−0.1480 (3)	0.13133 (16)	0.0331 (5)
H8B	0.379285	−0.172851	0.083366	0.040*
C9B	0.4325 (2)	−0.2415 (3)	0.18302 (19)	0.0416 (7)
H9B	0.440569	−0.327656	0.169875	0.050*
C10B	0.4565 (2)	−0.2081 (3)	0.25186 (19)	0.0447 (8)
H10B	0.480564	−0.271730	0.286887	0.054*
C11B	0.44597 (19)	−0.0799 (4)	0.27190 (15)	0.0408 (6)
C12B	0.4749 (3)	−0.0458 (5)	0.34383 (18)	0.0598 (11)
H12B	0.499686	−0.110313	0.378138	0.072*
C13B	0.4677 (3)	0.0782 (6)	0.36478 (18)	0.0675 (12)
H13B	0.488485	0.100209	0.413208	0.081*
C14B	0.4294 (3)	0.1732 (4)	0.31411 (18)	0.0557 (9)
H14B	0.423864	0.259353	0.328671	0.067*
C15B	0.3998 (2)	0.1431 (3)	0.24405 (15)	0.0378 (6)
H15B	0.373284	0.208679	0.210960	0.045*
C16B	0.40786 (17)	0.0161 (3)	0.21970 (14)	0.0312 (5)
C17B	0.18258 (14)	0.0260 (2)	0.07173 (12)	0.0240 (4)
C18B	0.17658 (17)	−0.0904 (3)	0.03616 (16)	0.0362 (6)
H18B	0.222145	−0.116942	0.020048	0.043*
C19B	0.10414 (19)	−0.1690 (3)	0.02384 (17)	0.0403 (6)
H19B	0.101668	−0.249542	0.000471	0.048*
C20B	0.03663 (18)	−0.1320 (3)	0.04485 (16)	0.0375 (6)
H20B	−0.013176	−0.184875	0.035055	0.045*
C21B	0.0420 (2)	−0.0165 (4)	0.0805 (2)	0.0458 (7)
H21B	−0.004282	0.010201	0.095651	0.055*
C22B	0.11428 (19)	0.0607 (3)	0.09440 (17)	0.0368 (6)
H22B	0.117436	0.138935	0.119927	0.044*
C23B	0.22633 (14)	0.2390 (2)	0.03590 (13)	0.0239 (4)
C24B	0.18545 (14)	0.2252 (2)	−0.03469 (12)	0.0231 (4)
H24B	0.170904	0.141171	−0.054186	0.028*
C25B	0.16467 (14)	0.3353 (2)	−0.07890 (13)	0.0245 (4)
C26B	0.12002 (16)	0.3252 (3)	−0.15184 (14)	0.0270 (5)
H26B	0.102173	0.243203	−0.173015	0.032*
C27B	0.10314 (16)	0.4359 (3)	−0.19113 (13)	0.0305 (5)
C28B	0.13093 (17)	0.5590 (3)	−0.16304 (16)	0.0325 (5)
H28B	0.120889	0.633205	−0.192502	0.039*
C29B	0.17314 (16)	0.5698 (3)	−0.09175 (16)	0.0321 (5)
H29B	0.191820	0.652478	−0.071822	0.039*
C30B	0.18881 (14)	0.4591 (2)	−0.04815 (14)	0.0270 (5)
C31B	0.24184 (15)	0.3705 (3)	0.06140 (14)	0.0284 (5)
C32B	0.2775 (3)	0.5082 (4)	0.1616 (2)	0.0605 (11)
H32D	0.312393	0.565497	0.143091	0.091*
H32E	0.302792	0.503171	0.212812	0.091*
H32F	0.218838	0.542620	0.149019	0.091*
C33B	0.64439 (16)	0.7125 (3)	0.11789 (12)	0.0275 (5)
C34B	0.68382 (16)	0.7862 (3)	0.18508 (12)	0.0262 (4)

H34B	0.740262	0.764391	0.214460	0.039 (9)*
C35B	0.64195 (16)	0.8809 (3)	0.20449 (13)	0.0270 (5)
H35B	0.583327	0.894093	0.177285	0.036 (9)*
C36B	0.67970 (16)	0.9671 (3)	0.26520 (14)	0.0294 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1A	0.0876 (3)	0.0599 (2)	0.0575 (2)	0.0371 (2)	0.0360 (2)	0.02961 (19)
O1A	0.0325 (9)	0.0275 (9)	0.0287 (8)	−0.0026 (7)	0.0171 (7)	−0.0036 (7)
O2A	0.0461 (12)	0.0492 (14)	0.0444 (12)	0.0007 (10)	0.0073 (10)	−0.0244 (11)
O3A	0.0314 (9)	0.0409 (11)	0.0239 (8)	−0.0039 (8)	−0.0006 (7)	0.0011 (8)
O4A	0.0513 (12)	0.0284 (9)	0.0209 (8)	−0.0064 (8)	0.0065 (8)	−0.0016 (7)
O5A	0.0397 (10)	0.0264 (9)	0.0319 (9)	−0.0017 (8)	0.0024 (8)	0.0014 (7)
O6A	0.0427 (11)	0.0284 (9)	0.0300 (9)	0.0029 (8)	−0.0027 (8)	−0.0046 (7)
N1A	0.0175 (8)	0.0260 (9)	0.0287 (9)	−0.0018 (7)	0.0061 (7)	0.0007 (8)
N2A	0.0254 (10)	0.0290 (11)	0.0671 (17)	−0.0033 (9)	0.0154 (11)	−0.0140 (12)
C1A	0.0206 (10)	0.0325 (12)	0.0239 (10)	0.0019 (9)	0.0089 (8)	−0.0032 (9)
C2A	0.0199 (9)	0.0269 (11)	0.0270 (11)	0.0006 (8)	0.0104 (8)	−0.0003 (8)
C3A	0.0206 (10)	0.0269 (11)	0.0309 (11)	−0.0004 (8)	0.0082 (9)	0.0004 (9)
C4A	0.0233 (11)	0.0325 (13)	0.0417 (14)	0.0030 (9)	0.0150 (10)	0.0121 (11)
C5A	0.0407 (15)	0.0458 (17)	0.0348 (14)	−0.0224 (13)	0.0125 (12)	−0.0103 (12)
C6A	0.0382 (15)	0.0335 (14)	0.071 (2)	0.0077 (12)	0.0326 (15)	0.0071 (15)
C7A	0.0209 (10)	0.0325 (13)	0.0342 (12)	0.0000 (9)	0.0129 (9)	0.0030 (9)
C8A	0.0437 (15)	0.0316 (13)	0.0409 (15)	0.0007 (11)	0.0237 (12)	0.0043 (11)
C9A	0.060 (2)	0.0337 (15)	0.060 (2)	0.0086 (14)	0.0344 (17)	0.0150 (14)
C10A	0.0437 (17)	0.0495 (19)	0.056 (2)	0.0096 (14)	0.0227 (15)	0.0258 (16)
C11A	0.0295 (12)	0.0538 (19)	0.0378 (14)	−0.0025 (12)	0.0098 (11)	0.0151 (13)
C12A	0.0464 (17)	0.072 (2)	0.0335 (14)	−0.0056 (18)	0.0047 (13)	0.0172 (17)
C13A	0.054 (2)	0.087 (3)	0.0293 (15)	−0.015 (2)	0.0020 (14)	0.0067 (17)
C14A	0.0524 (18)	0.059 (2)	0.0281 (13)	−0.0158 (16)	0.0079 (13)	−0.0067 (13)
C15A	0.0344 (13)	0.0455 (16)	0.0257 (12)	−0.0101 (12)	0.0080 (10)	−0.0030 (11)
C16A	0.0227 (10)	0.0420 (14)	0.0291 (11)	−0.0035 (10)	0.0072 (9)	0.0052 (11)
C17A	0.0212 (10)	0.0394 (14)	0.0241 (10)	0.0016 (9)	0.0095 (8)	0.0059 (9)
C18A	0.0367 (15)	0.0452 (18)	0.082 (3)	0.0018 (13)	0.0390 (17)	−0.0005 (17)
C19A	0.0343 (15)	0.062 (2)	0.087 (3)	0.0035 (15)	0.0370 (18)	0.012 (2)
C20A	0.0255 (12)	0.066 (2)	0.0507 (17)	−0.0085 (13)	0.0119 (12)	0.0184 (16)
C21A	0.0395 (15)	0.068 (2)	0.0419 (16)	−0.0241 (16)	0.0185 (13)	−0.0132 (16)
C22A	0.0339 (13)	0.064 (2)	0.0487 (16)	−0.0199 (14)	0.0249 (12)	−0.0228 (16)
C23A	0.0212 (10)	0.0268 (11)	0.0352 (12)	0.0016 (9)	0.0116 (9)	−0.0069 (9)
C24A	0.0231 (10)	0.0244 (11)	0.0346 (12)	0.0041 (8)	0.0132 (9)	−0.0011 (9)
C25A	0.0225 (10)	0.0277 (12)	0.0462 (14)	0.0036 (9)	0.0171 (10)	0.0020 (11)
C26A	0.0356 (13)	0.0339 (14)	0.0445 (15)	0.0123 (11)	0.0211 (12)	0.0072 (12)
C27A	0.0404 (15)	0.0391 (15)	0.0610 (19)	0.0167 (13)	0.0274 (14)	0.0155 (15)
C28A	0.0340 (14)	0.0332 (15)	0.081 (3)	0.0102 (12)	0.0274 (16)	0.0152 (16)
C29A	0.0277 (13)	0.0251 (13)	0.087 (3)	0.0011 (10)	0.0228 (15)	0.0017 (15)
C30A	0.0206 (10)	0.0264 (11)	0.0600 (17)	0.0009 (9)	0.0167 (11)	−0.0022 (12)
C31A	0.0233 (11)	0.0355 (14)	0.0487 (16)	−0.0014 (10)	0.0108 (11)	−0.0159 (12)

C32A	0.064 (2)	0.060 (2)	0.073 (3)	−0.016 (2)	0.029 (2)	−0.044 (2)
C33A	0.0292 (11)	0.0269 (11)	0.0209 (10)	0.0030 (9)	0.0053 (8)	−0.0026 (8)
C34A	0.0243 (10)	0.0285 (11)	0.0234 (10)	0.0001 (9)	0.0025 (8)	−0.0008 (9)
C35A	0.0249 (10)	0.0270 (11)	0.0244 (10)	0.0002 (9)	0.0020 (8)	−0.0009 (9)
C36A	0.0286 (11)	0.0251 (12)	0.0275 (11)	0.0021 (9)	0.0057 (9)	−0.0014 (8)
Br1B	0.0713 (2)	0.03731 (15)	0.03003 (13)	0.00847 (15)	0.01267 (13)	0.00635 (12)
O1B	0.0293 (8)	0.0296 (9)	0.0256 (8)	0.0064 (7)	0.0151 (7)	0.0038 (7)
O2B	0.0409 (10)	0.0350 (10)	0.0348 (10)	0.0019 (8)	0.0031 (8)	−0.0121 (8)
O3B	0.0293 (10)	0.0694 (16)	0.0297 (10)	0.0111 (10)	−0.0034 (8)	−0.0150 (10)
O4B	0.0356 (9)	0.0289 (9)	0.0230 (8)	0.0020 (7)	0.0084 (7)	−0.0004 (7)
O5B	0.0342 (10)	0.0326 (11)	0.0631 (14)	0.0041 (8)	0.0079 (10)	−0.0164 (10)
O6B	0.0472 (11)	0.0280 (9)	0.0306 (9)	0.0058 (9)	−0.0054 (8)	−0.0075 (9)
N1B	0.0164 (8)	0.0402 (12)	0.0255 (9)	−0.0009 (8)	0.0031 (7)	0.0079 (9)
N2B	0.0229 (9)	0.0247 (9)	0.0402 (12)	−0.0008 (8)	0.0063 (8)	−0.0054 (9)
C1B	0.0193 (9)	0.0272 (11)	0.0233 (10)	0.0022 (8)	0.0083 (8)	0.0006 (8)
C2B	0.0193 (9)	0.0284 (11)	0.0221 (10)	0.0043 (8)	0.0081 (8)	0.0057 (8)
C3B	0.0196 (10)	0.0290 (12)	0.0322 (11)	0.0016 (8)	0.0095 (9)	0.0081 (9)
C4B	0.0201 (10)	0.0364 (13)	0.0319 (12)	0.0031 (9)	0.0089 (9)	0.0121 (10)
C5B	0.0310 (13)	0.0404 (15)	0.0454 (16)	−0.0101 (11)	0.0064 (12)	0.0031 (13)
C6B	0.0250 (12)	0.059 (2)	0.0458 (16)	0.0085 (12)	0.0149 (11)	0.0128 (15)
C7B	0.0194 (9)	0.0311 (12)	0.0288 (11)	0.0012 (9)	0.0088 (8)	0.0057 (10)
C8B	0.0300 (12)	0.0300 (13)	0.0422 (14)	0.0041 (10)	0.0160 (11)	0.0092 (11)
C9B	0.0358 (14)	0.0357 (15)	0.0560 (18)	0.0083 (12)	0.0188 (13)	0.0171 (13)
C10B	0.0351 (14)	0.0460 (18)	0.0516 (18)	0.0116 (13)	0.0121 (13)	0.0282 (15)
C11B	0.0343 (13)	0.0529 (17)	0.0312 (12)	−0.0013 (13)	0.0055 (10)	0.0153 (14)
C12B	0.060 (2)	0.078 (3)	0.0307 (14)	0.004 (2)	0.0002 (14)	0.0205 (17)
C13B	0.077 (3)	0.088 (3)	0.0234 (14)	−0.006 (2)	−0.0024 (16)	0.0039 (17)
C14B	0.067 (2)	0.059 (2)	0.0308 (15)	−0.0105 (18)	0.0022 (15)	−0.0046 (15)
C15B	0.0381 (14)	0.0442 (16)	0.0267 (12)	−0.0045 (12)	0.0047 (11)	0.0014 (11)
C16B	0.0236 (11)	0.0400 (14)	0.0275 (11)	−0.0009 (10)	0.0052 (9)	0.0083 (10)
C17B	0.0207 (10)	0.0295 (11)	0.0225 (10)	0.0029 (8)	0.0081 (8)	0.0042 (8)
C18B	0.0282 (11)	0.0403 (15)	0.0452 (14)	−0.0070 (11)	0.0189 (11)	−0.0112 (12)
C19B	0.0354 (14)	0.0434 (16)	0.0445 (16)	−0.0130 (12)	0.0163 (12)	−0.0110 (13)
C20B	0.0278 (12)	0.0474 (17)	0.0382 (14)	−0.0103 (11)	0.0120 (11)	0.0046 (13)
C21B	0.0317 (13)	0.0533 (19)	0.063 (2)	−0.0026 (13)	0.0302 (14)	0.0012 (17)
C22B	0.0330 (13)	0.0365 (14)	0.0499 (16)	−0.0005 (11)	0.0259 (12)	−0.0019 (12)
C23B	0.0187 (9)	0.0231 (10)	0.0309 (11)	0.0022 (8)	0.0093 (8)	−0.0017 (8)
C24B	0.0193 (9)	0.0220 (10)	0.0277 (10)	0.0029 (8)	0.0074 (8)	−0.0002 (8)
C25B	0.0188 (9)	0.0242 (11)	0.0309 (11)	0.0030 (8)	0.0088 (8)	0.0014 (9)
C26B	0.0273 (11)	0.0247 (11)	0.0313 (12)	0.0028 (9)	0.0127 (9)	0.0021 (9)
C27B	0.0309 (11)	0.0313 (13)	0.0317 (11)	0.0055 (10)	0.0136 (9)	0.0061 (10)
C28B	0.0293 (12)	0.0269 (12)	0.0454 (15)	0.0029 (10)	0.0180 (11)	0.0089 (11)
C29B	0.0233 (11)	0.0232 (11)	0.0519 (16)	−0.0007 (9)	0.0151 (11)	0.0020 (11)
C30B	0.0177 (9)	0.0224 (11)	0.0410 (13)	0.0008 (8)	0.0096 (9)	0.0008 (9)
C31B	0.0203 (10)	0.0284 (12)	0.0345 (12)	0.0007 (9)	0.0063 (9)	−0.0061 (10)
C32B	0.072 (3)	0.044 (2)	0.051 (2)	−0.0037 (17)	0.0006 (18)	−0.0245 (16)
C33B	0.0273 (11)	0.0343 (13)	0.0196 (10)	0.0008 (9)	0.0059 (8)	−0.0007 (9)
C34B	0.0241 (10)	0.0299 (12)	0.0223 (10)	0.0010 (9)	0.0043 (8)	−0.0013 (9)

C35B	0.0242 (10)	0.0263 (11)	0.0277 (11)	0.0007 (9)	0.0048 (9)	0.0004 (9)
C36B	0.0295 (11)	0.0245 (11)	0.0330 (12)	0.0004 (9)	0.0085 (9)	−0.0015 (9)

Geometric parameters (Å, º)

Br1A—C27A	1.891 (4)	Br1B—C27B	1.902 (3)
O1A—C2A	1.425 (3)	O1B—C2B	1.425 (3)
O1A—H1AB	0.86 (4)	O1B—H1B	0.89 (4)
O2A—C31A	1.363 (4)	O2B—C31B	1.354 (3)
O2A—C32A	1.434 (4)	O2B—C32B	1.430 (4)
O3A—C33A	1.258 (3)	O3B—C33B	1.261 (3)
O4A—C33A	1.263 (3)	O4B—C33B	1.257 (3)
O5A—C36A	1.323 (3)	O5B—C36B	1.211 (3)
O5A—H5A	1.04 (4)	O6B—C36B	1.326 (3)
O6A—C36A	1.206 (3)	O6B—H6B	0.86 (5)
N1A—C5A	1.478 (3)	N1B—C6B	1.479 (4)
N1A—C6A	1.485 (4)	N1B—C5B	1.484 (4)
N1A—C4A	1.498 (3)	N1B—C4B	1.491 (3)
N1A—H1AN	0.98 (4)	N1B—H1BN	0.85 (4)
N2A—C31A	1.296 (4)	N2B—C31B	1.299 (4)
N2A—C30A	1.371 (4)	N2B—C30B	1.372 (4)
C1A—C23A	1.514 (4)	C1B—C23B	1.511 (3)
C1A—C17A	1.535 (3)	C1B—C17B	1.536 (3)
C1A—C2A	1.568 (3)	C1B—C2B	1.576 (3)
C1A—H1A	1.0000	C1B—H1BA	1.0000
C2A—C7A	1.535 (4)	C2B—C7B	1.539 (3)
C2A—C3A	1.551 (3)	C2B—C3B	1.548 (3)
C3A—C4A	1.535 (3)	C3B—C4B	1.530 (3)
C3A—H3AA	0.9900	C3B—H3BA	0.9900
C3A—H3AB	0.9900	C3B—H3BB	0.9900
C4A—H4AA	0.9900	C4B—H4BA	0.9900
C4A—H4AB	0.9900	C4B—H4BB	0.9900
C5A—H5AA	0.9800	C5B—H5BA	0.9800
C5A—H5AB	0.9800	C5B—H5BB	0.9800
C5A—H5AC	0.9800	C5B—H5BC	0.9800
C6A—H6AA	0.9800	C6B—H6BA	0.9800
C6A—H6AB	0.9800	C6B—H6BB	0.9800
C6A—H6AC	0.9800	C6B—H6BC	0.9800
C7A—C8A	1.381 (4)	C7B—C8B	1.378 (4)
C7A—C16A	1.437 (4)	C7B—C16B	1.434 (4)
C8A—C9A	1.406 (4)	C8B—C9B	1.403 (4)
C8A—H8A	0.9500	C8B—H8B	0.9500
C9A—C10A	1.355 (6)	C9B—C10B	1.357 (5)
C9A—H9A	0.9500	C9B—H9B	0.9500
C10A—C11A	1.417 (6)	C10B—C11B	1.411 (6)
C10A—H10A	0.9500	C10B—H10B	0.9500
C11A—C12A	1.416 (5)	C11B—C12B	1.414 (5)
C11A—C16A	1.432 (4)	C11B—C16B	1.432 (4)

C12A—C13A	1.351 (7)	C12B—C13B	1.365 (7)
C12A—H12A	0.9500	C12B—H12B	0.9500
C13A—C14A	1.406 (6)	C13B—C14B	1.407 (6)
C13A—H13A	0.9500	C13B—H13B	0.9500
C14A—C15A	1.369 (4)	C14B—C15B	1.370 (4)
C14A—H14A	0.9500	C14B—H14B	0.9500
C15A—C16A	1.430 (5)	C15B—C16B	1.421 (4)
C15A—H15A	0.9500	C15B—H15B	0.9500
C17A—C22A	1.371 (4)	C17B—C18B	1.387 (4)
C17A—C18A	1.387 (4)	C17B—C22B	1.392 (3)
C18A—C19A	1.391 (5)	C18B—C19B	1.395 (4)
C18A—H18A	0.9500	C18B—H18B	0.9500
C19A—C20A	1.357 (6)	C19B—C20B	1.367 (4)
C19A—H19A	0.9500	C19B—H19B	0.9500
C20A—C21A	1.357 (5)	C20B—C21B	1.380 (5)
C20A—H20A	0.9500	C20B—H20B	0.9500
C21A—C22A	1.403 (4)	C21B—C22B	1.381 (4)
C21A—H21A	0.9500	C21B—H21B	0.9500
C22A—H22A	0.9500	C22B—H22B	0.9500
C23A—C24A	1.366 (4)	C23B—C24B	1.367 (3)
C23A—C31A	1.443 (4)	C23B—C31B	1.444 (3)
C24A—C25A	1.415 (4)	C24B—C25B	1.415 (3)
C24A—H24A	0.9500	C24B—H24B	0.9500
C25A—C30A	1.413 (4)	C25B—C26B	1.415 (4)
C25A—C26A	1.414 (4)	C25B—C30B	1.419 (4)
C26A—C27A	1.370 (4)	C26B—C27B	1.366 (4)
C26A—H26A	0.9500	C26B—H26B	0.9500
C27A—C28A	1.397 (5)	C27B—C28B	1.404 (4)
C28A—C29A	1.366 (6)	C28B—C29B	1.380 (4)
C28A—H28A	0.9500	C28B—H28B	0.9500
C29A—C30A	1.417 (5)	C29B—C30B	1.413 (4)
C29A—H29A	0.9500	C29B—H29B	0.9500
C32A—H32A	0.9800	C32B—H32D	0.9800
C32A—H32B	0.9800	C32B—H32E	0.9800
C32A—H32C	0.9800	C32B—H32F	0.9800
C33A—C34A	1.498 (3)	C33B—C34B	1.502 (3)
C34A—C35A	1.320 (4)	C34B—C35B	1.327 (4)
C34A—H34A	0.9500	C34B—H34B	0.9500
C35A—C36A	1.498 (3)	C35B—C36B	1.476 (4)
C35A—H35A	0.9500	C35B—H35B	0.9500

C2A—O1A—H1AB	112 (3)	C2B—O1B—H1B	111 (3)
C31A—O2A—C32A	117.6 (3)	C31B—O2B—C32B	117.4 (3)
C36A—O5A—H5A	107 (3)	C36B—O6B—H6B	105 (3)
C5A—N1A—C6A	110.7 (2)	C6B—N1B—C5B	109.8 (2)
C5A—N1A—C4A	112.5 (2)	C6B—N1B—C4B	110.6 (2)
C6A—N1A—C4A	110.8 (2)	C5B—N1B—C4B	113.6 (2)
C5A—N1A—H1AN	110 (2)	C6B—N1B—H1BN	104 (3)

C6A—N1A—H1AN	104 (2)	C5B—N1B—H1BN	109 (3)
C4A—N1A—H1AN	108 (2)	C4B—N1B—H1BN	109 (3)
C31A—N2A—C30A	117.5 (3)	C31B—N2B—C30B	117.4 (2)
C23A—C1A—C17A	109.67 (19)	C23B—C1B—C17B	109.78 (18)
C23A—C1A—C2A	111.83 (19)	C23B—C1B—C2B	111.04 (18)
C17A—C1A—C2A	116.8 (2)	C17B—C1B—C2B	116.5 (2)
C23A—C1A—H1A	105.9	C23B—C1B—H1BA	106.3
C17A—C1A—H1A	105.9	C17B—C1B—H1BA	106.3
C2A—C1A—H1A	105.9	C2B—C1B—H1BA	106.3
O1A—C2A—C7A	107.7 (2)	O1B—C2B—C7B	107.4 (2)
O1A—C2A—C3A	106.58 (19)	O1B—C2B—C3B	106.19 (18)
C7A—C2A—C3A	110.6 (2)	C7B—C2B—C3B	111.09 (19)
O1A—C2A—C1A	109.90 (19)	O1B—C2B—C1B	110.36 (18)
C7A—C2A—C1A	109.93 (19)	C7B—C2B—C1B	110.11 (18)
C3A—C2A—C1A	112.0 (2)	C3B—C2B—C1B	111.5 (2)
C4A—C3A—C2A	109.8 (2)	C4B—C3B—C2B	110.0 (2)
C4A—C3A—H3AA	109.7	C4B—C3B—H3BA	109.7
C2A—C3A—H3AA	109.7	C2B—C3B—H3BA	109.7
C4A—C3A—H3AB	109.7	C4B—C3B—H3BB	109.7
C2A—C3A—H3AB	109.7	C2B—C3B—H3BB	109.7
H3AA—C3A—H3AB	108.2	H3BA—C3B—H3BB	108.2
N1A—C4A—C3A	113.3 (2)	N1B—C4B—C3B	112.3 (2)
N1A—C4A—H4AA	108.9	N1B—C4B—H4BA	109.2
C3A—C4A—H4AA	108.9	C3B—C4B—H4BA	109.2
N1A—C4A—H4AB	108.9	N1B—C4B—H4BB	109.2
C3A—C4A—H4AB	108.9	C3B—C4B—H4BB	109.2
H4AA—C4A—H4AB	107.7	H4BA—C4B—H4BB	107.9
N1A—C5A—H5AA	109.5	N1B—C5B—H5BA	109.5
N1A—C5A—H5AB	109.5	N1B—C5B—H5BB	109.5
H5AA—C5A—H5AB	109.5	H5BA—C5B—H5BB	109.5
N1A—C5A—H5AC	109.5	N1B—C5B—H5BC	109.5
H5AA—C5A—H5AC	109.5	H5BA—C5B—H5BC	109.5
H5AB—C5A—H5AC	109.5	H5BB—C5B—H5BC	109.5
N1A—C6A—H6AA	109.5	N1B—C6B—H6BA	109.5
N1A—C6A—H6AB	109.5	N1B—C6B—H6BB	109.5
H6AA—C6A—H6AB	109.5	H6BA—C6B—H6BB	109.5
N1A—C6A—H6AC	109.5	N1B—C6B—H6BC	109.5
H6AA—C6A—H6AC	109.5	H6BA—C6B—H6BC	109.5
H6AB—C6A—H6AC	109.5	H6BB—C6B—H6BC	109.5
C8A—C7A—C16A	118.2 (3)	C8B—C7B—C16B	118.6 (2)
C8A—C7A—C2A	117.8 (2)	C8B—C7B—C2B	117.9 (2)
C16A—C7A—C2A	123.9 (2)	C16B—C7B—C2B	123.3 (2)
C7A—C8A—C9A	122.6 (3)	C7B—C8B—C9B	122.7 (3)
C7A—C8A—H8A	118.7	C7B—C8B—H8B	118.6
C9A—C8A—H8A	118.7	C9B—C8B—H8B	118.6
C10A—C9A—C8A	120.3 (3)	C10B—C9B—C8B	119.7 (3)
C10A—C9A—H9A	119.9	C10B—C9B—H9B	120.2
C8A—C9A—H9A	119.9	C8B—C9B—H9B	120.2

C9A—C10A—C11A	120.1 (3)	C9B—C10B—C11B	120.6 (3)
C9A—C10A—H10A	120.0	C9B—C10B—H10B	119.7
C11A—C10A—H10A	120.0	C11B—C10B—H10B	119.7
C12A—C11A—C10A	119.7 (3)	C10B—C11B—C12B	119.7 (3)
C12A—C11A—C16A	120.0 (3)	C10B—C11B—C16B	120.3 (3)
C10A—C11A—C16A	120.3 (3)	C12B—C11B—C16B	120.0 (4)
C13A—C12A—C11A	121.3 (3)	C13B—C12B—C11B	121.1 (3)
C13A—C12A—H12A	119.4	C13B—C12B—H12B	119.5
C11A—C12A—H12A	119.4	C11B—C12B—H12B	119.5
C12A—C13A—C14A	119.8 (3)	C12B—C13B—C14B	119.5 (3)
C12A—C13A—H13A	120.1	C12B—C13B—H13B	120.3
C14A—C13A—H13A	120.1	C14B—C13B—H13B	120.3
C15A—C14A—C13A	120.9 (4)	C15B—C14B—C13B	120.9 (4)
C15A—C14A—H14A	119.6	C15B—C14B—H14B	119.6
C13A—C14A—H14A	119.6	C13B—C14B—H14B	119.6
C14A—C15A—C16A	121.3 (3)	C14B—C15B—C16B	121.6 (3)
C14A—C15A—H15A	119.3	C14B—C15B—H15B	119.2
C16A—C15A—H15A	119.3	C16B—C15B—H15B	119.2
C15A—C16A—C11A	116.6 (3)	C15B—C16B—C11B	116.9 (3)
C15A—C16A—C7A	124.8 (3)	C15B—C16B—C7B	125.0 (2)
C11A—C16A—C7A	118.5 (3)	C11B—C16B—C7B	118.1 (3)
C22A—C17A—C18A	117.2 (3)	C18B—C17B—C22B	117.7 (2)
C22A—C17A—C1A	126.7 (2)	C18B—C17B—C1B	126.6 (2)
C18A—C17A—C1A	116.0 (3)	C22B—C17B—C1B	115.6 (2)
C17A—C18A—C19A	121.2 (4)	C17B—C18B—C19B	120.5 (3)
C17A—C18A—H18A	119.4	C17B—C18B—H18B	119.8
C19A—C18A—H18A	119.4	C19B—C18B—H18B	119.8
C20A—C19A—C18A	120.7 (3)	C20B—C19B—C18B	121.1 (3)
C20A—C19A—H19A	119.6	C20B—C19B—H19B	119.4
C18A—C19A—H19A	119.6	C18B—C19B—H19B	119.4
C21A—C20A—C19A	119.1 (3)	C19B—C20B—C21B	118.9 (3)
C21A—C20A—H20A	120.5	C19B—C20B—H20B	120.6
C19A—C20A—H20A	120.5	C21B—C20B—H20B	120.6
C20A—C21A—C22A	120.7 (4)	C20B—C21B—C22B	120.5 (3)
C20A—C21A—H21A	119.6	C20B—C21B—H21B	119.8
C22A—C21A—H21A	119.6	C22B—C21B—H21B	119.8
C17A—C22A—C21A	121.1 (3)	C21B—C22B—C17B	121.3 (3)
C17A—C22A—H22A	119.5	C21B—C22B—H22B	119.3
C21A—C22A—H22A	119.5	C17B—C22B—H22B	119.3
C24A—C23A—C31A	115.7 (3)	C24B—C23B—C31B	115.9 (2)
C24A—C23A—C1A	123.6 (2)	C24B—C23B—C1B	123.5 (2)
C31A—C23A—C1A	120.7 (2)	C31B—C23B—C1B	120.5 (2)
C23A—C24A—C25A	120.7 (2)	C23B—C24B—C25B	120.4 (2)
C23A—C24A—H24A	119.7	C23B—C24B—H24B	119.8
C25A—C24A—H24A	119.7	C25B—C24B—H24B	119.8
C30A—C25A—C26A	119.5 (3)	C24B—C25B—C26B	122.1 (2)
C30A—C25A—C24A	118.2 (3)	C24B—C25B—C30B	118.2 (2)
C26A—C25A—C24A	122.3 (3)	C26B—C25B—C30B	119.7 (2)

C27A—C26A—C25A	118.9 (3)	C27B—C26B—C25B	118.6 (2)
C27A—C26A—H26A	120.6	C27B—C26B—H26B	120.7
C25A—C26A—H26A	120.6	C25B—C26B—H26B	120.7
C26A—C27A—C28A	122.8 (3)	C26B—C27B—C28B	123.1 (2)
C26A—C27A—Br1A	119.0 (3)	C26B—C27B—Br1B	118.9 (2)
C28A—C27A—Br1A	118.2 (3)	C28B—C27B—Br1B	118.0 (2)
C29A—C28A—C27A	118.6 (3)	C29B—C28B—C27B	118.6 (2)
C29A—C28A—H28A	120.7	C29B—C28B—H28B	120.7
C27A—C28A—H28A	120.7	C27B—C28B—H28B	120.7
C28A—C29A—C30A	121.3 (3)	C28B—C29B—C30B	120.6 (3)
C28A—C29A—H29A	119.4	C28B—C29B—H29B	119.7
C30A—C29A—H29A	119.4	C30B—C29B—H29B	119.7
N2A—C30A—C25A	121.7 (3)	N2B—C30B—C29B	119.0 (2)
N2A—C30A—C29A	119.4 (3)	N2B—C30B—C25B	121.8 (2)
C25A—C30A—C29A	118.9 (3)	C29B—C30B—C25B	119.3 (2)
N2A—C31A—O2A	120.2 (3)	N2B—C31B—O2B	119.4 (2)
N2A—C31A—C23A	126.1 (3)	N2B—C31B—C23B	126.0 (2)
O2A—C31A—C23A	113.7 (3)	O2B—C31B—C23B	114.7 (2)
O2A—C32A—H32A	109.5	O2B—C32B—H32D	109.5
O2A—C32A—H32B	109.5	O2B—C32B—H32E	109.5
H32A—C32A—H32B	109.5	H32D—C32B—H32E	109.5
O2A—C32A—H32C	109.5	O2B—C32B—H32F	109.5
H32A—C32A—H32C	109.5	H32D—C32B—H32F	109.5
H32B—C32A—H32C	109.5	H32E—C32B—H32F	109.5
O3A—C33A—O4A	124.3 (2)	O4B—C33B—O3B	125.1 (2)
O3A—C33A—C34A	118.4 (2)	O4B—C33B—C34B	117.6 (2)
O4A—C33A—C34A	117.4 (2)	O3B—C33B—C34B	117.3 (2)
C35A—C34A—C33A	123.4 (2)	C35B—C34B—C33B	121.8 (2)
C35A—C34A—H34A	118.3	C35B—C34B—H34B	119.1
C33A—C34A—H34A	118.3	C33B—C34B—H34B	119.1
C34A—C35A—C36A	120.6 (2)	C34B—C35B—C36B	124.8 (2)
C34A—C35A—H35A	119.7	C34B—C35B—H35B	117.6
C36A—C35A—H35A	119.7	C36B—C35B—H35B	117.6
O6A—C36A—O5A	124.6 (2)	O5B—C36B—O6B	123.8 (3)
O6A—C36A—C35A	123.3 (2)	O5B—C36B—C35B	121.5 (2)
O5A—C36A—C35A	112.1 (2)	O6B—C36B—C35B	114.6 (2)

C23A—C1A—C2A—O1A	−76.0 (3)	C23B—C1B—C2B—O1B	−73.1 (2)
C17A—C1A—C2A—O1A	51.5 (3)	C17B—C1B—C2B—O1B	53.6 (3)
C23A—C1A—C2A—C7A	165.5 (2)	C23B—C1B—C2B—C7B	168.5 (2)
C17A—C1A—C2A—C7A	−67.0 (3)	C17B—C1B—C2B—C7B	−64.8 (3)
C23A—C1A—C2A—C3A	42.2 (3)	C23B—C1B—C2B—C3B	44.7 (3)
C17A—C1A—C2A—C3A	169.7 (2)	C17B—C1B—C2B—C3B	171.38 (19)
O1A—C2A—C3A—C4A	−63.8 (3)	O1B—C2B—C3B—C4B	−64.8 (2)
C7A—C2A—C3A—C4A	53.1 (3)	C7B—C2B—C3B—C4B	51.7 (3)
C1A—C2A—C3A—C4A	176.0 (2)	C1B—C2B—C3B—C4B	174.92 (19)
C5A—N1A—C4A—C3A	82.3 (3)	C6B—N1B—C4B—C3B	−168.1 (2)
C6A—N1A—C4A—C3A	−153.2 (2)	C5B—N1B—C4B—C3B	67.9 (3)

C2A—C3A—C4A—N1A	137.2 (2)	C2B—C3B—C4B—N1B	133.7 (2)
O1A—C2A—C7A—C8A	−0.2 (3)	O1B—C2B—C7B—C8B	−2.6 (3)
C3A—C2A—C7A—C8A	−116.3 (3)	C3B—C2B—C7B—C8B	−118.3 (2)
C1A—C2A—C7A—C8A	119.5 (2)	C1B—C2B—C7B—C8B	117.6 (2)
O1A—C2A—C7A—C16A	177.1 (2)	O1B—C2B—C7B—C16B	173.7 (2)
C3A—C2A—C7A—C16A	61.0 (3)	C3B—C2B—C7B—C16B	58.0 (3)
C1A—C2A—C7A—C16A	−63.1 (3)	C1B—C2B—C7B—C16B	−66.1 (3)
C16A—C7A—C8A—C9A	0.8 (4)	C16B—C7B—C8B—C9B	0.6 (4)
C2A—C7A—C8A—C9A	178.3 (3)	C2B—C7B—C8B—C9B	177.1 (2)
C7A—C8A—C9A—C10A	0.2 (5)	C7B—C8B—C9B—C10B	−0.1 (4)
C8A—C9A—C10A—C11A	−0.8 (5)	C8B—C9B—C10B—C11B	−1.0 (5)
C9A—C10A—C11A—C12A	−178.6 (3)	C9B—C10B—C11B—C12B	−177.2 (3)
C9A—C10A—C11A—C16A	0.5 (5)	C9B—C10B—C11B—C16B	1.5 (5)
C10A—C11A—C12A—C13A	179.2 (4)	C10B—C11B—C12B—C13B	178.2 (4)
C16A—C11A—C12A—C13A	0.2 (5)	C16B—C11B—C12B—C13B	−0.5 (6)
C11A—C12A—C13A—C14A	0.3 (6)	C11B—C12B—C13B—C14B	1.3 (7)
C12A—C13A—C14A—C15A	−0.7 (6)	C12B—C13B—C14B—C15B	−0.6 (7)
C13A—C14A—C15A—C16A	0.7 (5)	C13B—C14B—C15B—C16B	−1.0 (6)
C14A—C15A—C16A—C11A	−0.2 (4)	C14B—C15B—C16B—C11B	1.8 (5)
C14A—C15A—C16A—C7A	−179.9 (3)	C14B—C15B—C16B—C7B	−176.9 (3)
C12A—C11A—C16A—C15A	−0.2 (4)	C10B—C11B—C16B—C15B	−179.8 (3)
C10A—C11A—C16A—C15A	−179.3 (3)	C12B—C11B—C16B—C15B	−1.1 (4)
C12A—C11A—C16A—C7A	179.5 (3)	C10B—C11B—C16B—C7B	−0.9 (4)
C10A—C11A—C16A—C7A	0.4 (4)	C12B—C11B—C16B—C7B	177.8 (3)
C8A—C7A—C16A—C15A	178.6 (3)	C8B—C7B—C16B—C15B	178.6 (3)
C2A—C7A—C16A—C15A	1.3 (4)	C2B—C7B—C16B—C15B	2.4 (4)
C8A—C7A—C16A—C11A	−1.1 (4)	C8B—C7B—C16B—C11B	−0.1 (4)
C2A—C7A—C16A—C11A	−178.4 (2)	C2B—C7B—C16B—C11B	−176.4 (2)
C23A—C1A—C17A—C22A	108.7 (3)	C23B—C1B—C17B—C18B	103.0 (3)
C2A—C1A—C17A—C22A	−19.8 (4)	C2B—C1B—C17B—C18B	−24.3 (3)
C23A—C1A—C17A—C18A	−68.6 (3)	C23B—C1B—C17B—C22B	−73.6 (3)
C2A—C1A—C17A—C18A	162.8 (3)	C2B—C1B—C17B—C22B	159.1 (2)
C22A—C17A—C18A—C19A	−1.8 (5)	C22B—C17B—C18B—C19B	0.1 (4)
C1A—C17A—C18A—C19A	175.8 (3)	C1B—C17B—C18B—C19B	−176.4 (3)
C17A—C18A—C19A—C20A	2.3 (6)	C17B—C18B—C19B—C20B	1.6 (5)
C18A—C19A—C20A—C21A	−0.7 (6)	C18B—C19B—C20B—C21B	−1.8 (5)
C19A—C20A—C21A—C22A	−1.3 (6)	C19B—C20B—C21B—C22B	0.3 (5)
C18A—C17A—C22A—C21A	−0.2 (5)	C20B—C21B—C22B—C17B	1.4 (5)
C1A—C17A—C22A—C21A	−177.6 (3)	C18B—C17B—C22B—C21B	−1.6 (4)
C20A—C21A—C22A—C17A	1.8 (6)	C1B—C17B—C22B—C21B	175.3 (3)
C17A—C1A—C23A—C24A	−57.4 (3)	C17B—C1B—C23B—C24B	−46.9 (3)
C2A—C1A—C23A—C24A	73.8 (3)	C2B—C1B—C23B—C24B	83.4 (3)
C17A—C1A—C23A—C31A	122.5 (2)	C17B—C1B—C23B—C31B	133.8 (2)
C2A—C1A—C23A—C31A	−106.3 (3)	C2B—C1B—C23B—C31B	−95.9 (3)
C31A—C23A—C24A—C25A	2.7 (3)	C31B—C23B—C24B—C25B	3.2 (3)
C1A—C23A—C24A—C25A	−177.3 (2)	C1B—C23B—C24B—C25B	−176.1 (2)
C23A—C24A—C25A—C30A	−0.3 (3)	C23B—C24B—C25B—C26B	−178.0 (2)
C23A—C24A—C25A—C26A	−179.3 (2)	C23B—C24B—C25B—C30B	1.2 (3)

C30A—C25A—C26A—C27A	1.8 (4)	C24B—C25B—C26B—C27B	−179.3 (2)
C24A—C25A—C26A—C27A	−179.1 (3)	C30B—C25B—C26B—C27B	1.6 (3)
C25A—C26A—C27A—C28A	0.8 (4)	C25B—C26B—C27B—C28B	2.5 (4)
C25A—C26A—C27A—Br1A	−178.82 (19)	C25B—C26B—C27B—Br1B	−176.19 (17)
C26A—C27A—C28A—C29A	−2.2 (4)	C26B—C27B—C28B—C29B	−3.7 (4)
Br1A—C27A—C28A—C29A	177.4 (2)	Br1B—C27B—C28B—C29B	175.05 (19)
C27A—C28A—C29A—C30A	0.9 (4)	C27B—C28B—C29B—C30B	0.7 (4)
C31A—N2A—C30A—C25A	1.5 (4)	C31B—N2B—C30B—C29B	−178.3 (2)
C31A—N2A—C30A—C29A	−178.4 (2)	C31B—N2B—C30B—C25B	2.3 (3)
C26A—C25A—C30A—N2A	177.1 (2)	C28B—C29B—C30B—N2B	−176.1 (2)
C24A—C25A—C30A—N2A	−2.1 (4)	C28B—C29B—C30B—C25B	3.3 (4)
C26A—C25A—C30A—C29A	−3.0 (3)	C24B—C25B—C30B—N2B	−4.2 (3)
C24A—C25A—C30A—C29A	177.9 (2)	C26B—C25B—C30B—N2B	175.0 (2)
C28A—C29A—C30A—N2A	−178.4 (3)	C24B—C25B—C30B—C29B	176.4 (2)
C28A—C29A—C30A—C25A	1.7 (4)	C26B—C25B—C30B—C29B	−4.4 (3)
C30A—N2A—C31A—O2A	−178.0 (2)	C30B—N2B—C31B—O2B	−176.9 (2)
C30A—N2A—C31A—C23A	1.3 (4)	C30B—N2B—C31B—C23B	2.6 (4)
C32A—O2A—C31A—N2A	14.6 (4)	C32B—O2B—C31B—N2B	10.6 (4)
C32A—O2A—C31A—C23A	−164.7 (3)	C32B—O2B—C31B—C23B	−169.0 (3)
C24A—C23A—C31A—N2A	−3.5 (4)	C24B—C23B—C31B—N2B	−5.4 (4)
C1A—C23A—C31A—N2A	176.6 (2)	C1B—C23B—C31B—N2B	173.9 (2)
C24A—C23A—C31A—O2A	175.9 (2)	C24B—C23B—C31B—O2B	174.1 (2)
C1A—C23A—C31A—O2A	−4.1 (3)	C1B—C23B—C31B—O2B	−6.6 (3)
O3A—C33A—C34A—C35A	6.7 (4)	O4B—C33B—C34B—C35B	−178.3 (3)
O4A—C33A—C34A—C35A	−173.8 (3)	O3B—C33B—C34B—C35B	3.8 (4)
C33A—C34A—C35A—C36A	−176.1 (2)	C33B—C34B—C35B—C36B	−172.7 (2)
C34A—C35A—C36A—O6A	−30.1 (4)	C34B—C35B—C36B—O5B	161.3 (3)
C34A—C35A—C36A—O5A	148.2 (3)	C34B—C35B—C36B—O6B	−16.6 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1*AB*···O4*A*	0.86 (4)	1.88 (4)	2.699 (3)	159 (4)
O5*A*—H5*A*···O4*B*	1.04 (4)	1.58 (4)	2.603 (3)	169 (4)
N1*A*—H1*AN*···O3*A*	0.98 (4)	1.68 (4)	2.641 (3)	163 (3)
C1*A*—H1*A*···O2*A*	1.00	2.23	2.767 (3)	112
C3*A*—H3*AB*···Br1*B*^{i}	0.99	3.01	3.850 (3)	143
C5*A*—H5*AA*···O1*A*^{ii}	0.98	2.58	3.501 (3)	157
C5*A*—H5*AC*···Br1*B*^{i}	0.98	2.82	3.612 (3)	139
C26*A*—H26*A*···O5*B*^{iii}	0.95	2.54	3.459 (4)	163
C34*A*—H34*A*···O5*B*^{iii}	0.95	2.52	3.379 (3)	151
O1*B*—H1*B*···O4*B*^{iv}	0.89 (4)	1.88 (4)	2.741 (3)	160 (4)
O6*B*—H6*B*···O4*A*^{v}	0.86 (5)	1.80 (5)	2.625 (3)	159 (4)
N1*B*—H1*BN*···O3*B*^{iv}	0.85 (4)	1.83 (4)	2.632 (3)	158 (4)
C5*B*—H5*BA*···O1*B*^{vi}	0.98	2.42	3.337 (4)	156

C5*B*—H5*BB*···Br1*A*	0.98	2.88	3.468 (3)	119
C5*B*—H5*BB*···O6*A*	0.98	2.42	3.287 (4)	148

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

3-Benzyl-6-bromo-2-methoxyquinoline (3)

Crystal data

$C_{17}H_{14}BrNO$
M_r = 328.20
Orthorhombic, $P2_12_12_1$
a = 4.3606 (6) Å
b = 10.820 (2) Å
c = 29.886 (11) Å
V = 1410.1 (6) Å^3
Z = 4
F(000) = 664
D_x = 1.546 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 9920 reflections
θ = 2.3–32.7°
μ = 2.91 mm^{-1}
T = 150 K
Needle, colourless
0.41 × 0.06 × 0.05 mm

Data collection

Bruker D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD)
Radiation source: fine focus sealed tube X-ray source
Triumph curved graphite crystal monochromator
Detector resolution: 7.4074 pixels mm^{-1}
ω and phi scans
Absorption correction: multi-scan (SADABS2016; Krause *et al.*, 2015)
T_{min} = 0.658, T_{max} = 0.747
28030 measured reflections
5125 independent reflections
4504 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 33.1°, θ_{min} = 2.8°
h = −6→5
k = −16→16
l = −45→40

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.057
S = 1.05
5125 reflections
196 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
Only H-atom displacement parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Absolute structure: Flack *x* determined using 1685 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: −0.011 (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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.33234 (5)	1.09654 (2)	0.10466 (2)	0.03579 (6)

O1	0.9170 (3)	0.38638 (11)	0.08083 (4)	0.0283 (3)
N1	0.6613 (3)	0.56540 (12)	0.06323 (4)	0.0214 (2)
C1	0.8442 (4)	0.50555 (14)	0.09018 (5)	0.0214 (3)
C2	0.9779 (3)	0.55404 (15)	0.13019 (6)	0.0206 (3)
C3	0.9145 (3)	0.67422 (16)	0.14000 (6)	0.0212 (3)
H3	1.003058	0.711254	0.165763	0.027 (5)*
C4	0.7166 (3)	0.74501 (15)	0.11205 (5)	0.0191 (3)
C5	0.6405 (4)	0.86982 (15)	0.12085 (6)	0.0227 (3)
H5	0.724285	0.911329	0.146058	0.028 (5)*
C6	0.4437 (4)	0.92991 (14)	0.09234 (6)	0.0236 (3)
C7	0.3168 (4)	0.87254 (15)	0.05471 (6)	0.0247 (3)
H7	0.181534	0.916658	0.035583	0.034 (6)*
C8	0.3902 (4)	0.75158 (16)	0.04576 (6)	0.0229 (3)
H8	0.303981	0.711802	0.020373	0.031 (5)*
C9	0.5927 (3)	0.68571 (14)	0.07392 (5)	0.0192 (3)
C10	0.7927 (6)	0.33696 (18)	0.04034 (7)	0.0386 (5)
H10A	0.873753	0.383151	0.014732	0.045 (7)*
H10B	0.568653	0.344120	0.040923	0.051 (8)*
H10C	0.850322	0.249746	0.037603	0.048 (7)*
C11	1.1699 (4)	0.47323 (16)	0.16067 (6)	0.0240 (3)
H11A	1.325806	0.524748	0.175900	0.034 (6)*
H11B	1.278720	0.410507	0.142523	0.049 (7)*
C12	0.9739 (3)	0.40881 (15)	0.19546 (5)	0.0205 (3)
C13	0.8353 (4)	0.29555 (14)	0.18642 (6)	0.0246 (3)
H13	0.866613	0.256921	0.158245	0.022 (5)*
C14	0.6512 (4)	0.23861 (15)	0.21838 (6)	0.0294 (3)
H14	0.556300	0.161713	0.211782	0.055 (8)*
C15	0.6057 (4)	0.29308 (18)	0.25961 (7)	0.0310 (4)
H15	0.481362	0.253555	0.281437	0.035 (6)*
C16	0.7424 (4)	0.40577 (18)	0.26897 (6)	0.0302 (4)
H16	0.711861	0.443543	0.297316	0.035 (6)*
C17	0.9236 (4)	0.46362 (16)	0.23709 (6)	0.0267 (4)
H17	1.014500	0.541330	0.243665	0.051 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.04286 (10)	0.02454 (8)	0.03998 (11)	0.00613 (8)	−0.00053 (9)	−0.00279 (8)
O1	0.0374 (6)	0.0241 (6)	0.0233 (6)	0.0055 (5)	−0.0016 (5)	−0.0014 (5)
N1	0.0230 (6)	0.0238 (6)	0.0175 (6)	0.0004 (5)	−0.0001 (6)	−0.0002 (5)
C1	0.0220 (6)	0.0235 (7)	0.0188 (7)	0.0006 (6)	0.0033 (6)	0.0004 (5)
C2	0.0155 (6)	0.0279 (7)	0.0185 (7)	−0.0020 (5)	0.0015 (5)	0.0044 (6)
C3	0.0180 (6)	0.0278 (8)	0.0178 (7)	−0.0033 (5)	0.0000 (5)	0.0014 (6)
C4	0.0177 (6)	0.0243 (7)	0.0153 (7)	−0.0016 (5)	0.0025 (5)	0.0010 (5)
C5	0.0237 (7)	0.0254 (7)	0.0190 (7)	−0.0023 (6)	0.0022 (6)	−0.0016 (5)
C6	0.0244 (7)	0.0221 (7)	0.0242 (8)	0.0006 (5)	0.0061 (6)	0.0006 (6)
C7	0.0241 (7)	0.0286 (7)	0.0215 (8)	0.0026 (6)	0.0024 (7)	0.0049 (6)
C8	0.0229 (7)	0.0295 (8)	0.0164 (7)	−0.0005 (6)	−0.0005 (5)	0.0004 (6)

C9	0.0182 (6)	0.0234 (7)	0.0159 (7)	−0.0011 (5)	0.0030 (5)	0.0017 (6)
C10	0.0623 (13)	0.0286 (9)	0.0250 (9)	0.0078 (9)	−0.0066 (9)	−0.0059 (7)
C11	0.0165 (6)	0.0319 (8)	0.0237 (8)	0.0022 (6)	−0.0013 (7)	0.0036 (6)
C12	0.0169 (6)	0.0242 (7)	0.0205 (7)	0.0047 (6)	−0.0025 (5)	0.0039 (6)
C13	0.0252 (7)	0.0236 (7)	0.0251 (8)	0.0041 (7)	−0.0031 (7)	0.0005 (6)
C14	0.0300 (8)	0.0236 (7)	0.0345 (9)	−0.0001 (7)	−0.0037 (8)	0.0073 (7)
C15	0.0275 (8)	0.0345 (9)	0.0311 (9)	0.0036 (7)	0.0018 (7)	0.0131 (7)
C16	0.0335 (8)	0.0354 (9)	0.0216 (8)	0.0077 (7)	0.0013 (6)	0.0021 (7)
C17	0.0296 (8)	0.0260 (8)	0.0246 (9)	0.0011 (6)	−0.0038 (6)	−0.0011 (7)

Geometric parameters (Å, º)

Br1—C6	1.9030 (16)	C8—H8	0.9500
O1—C1	1.3570 (19)	C10—H10A	0.9800
O1—C10	1.430 (2)	C10—H10B	0.9800
N1—C1	1.305 (2)	C10—H10C	0.9800
N1—C9	1.373 (2)	C11—C12	1.516 (2)
C1—C2	1.430 (2)	C11—H11A	0.9900
C2—C3	1.361 (2)	C11—H11B	0.9900
C2—C11	1.515 (2)	C12—C13	1.393 (2)
C3—C4	1.424 (2)	C12—C17	1.396 (2)
C3—H3	0.9500	C13—C14	1.391 (3)
C4—C9	1.415 (2)	C13—H13	0.9500
C4—C5	1.415 (2)	C14—C15	1.380 (3)
C5—C6	1.373 (2)	C14—H14	0.9500
C5—H5	0.9500	C15—C16	1.386 (3)
C6—C7	1.399 (3)	C15—H15	0.9500
C7—C8	1.374 (2)	C16—C17	1.387 (3)
C7—H7	0.9500	C16—H16	0.9500
C8—C9	1.413 (2)	C17—H17	0.9500
C1—O1—C10	116.15 (14)	O1—C10—H10B	109.5
C1—N1—C9	117.37 (14)	H10A—C10—H10B	109.5
N1—C1—O1	119.16 (14)	O1—C10—H10C	109.5
N1—C1—C2	125.67 (15)	H10A—C10—H10C	109.5
O1—C1—C2	115.16 (14)	H10B—C10—H10C	109.5
C3—C2—C1	116.59 (15)	C2—C11—C12	111.49 (13)
C3—C2—C11	122.27 (16)	C2—C11—H11A	109.3
C1—C2—C11	121.09 (15)	C12—C11—H11A	109.3
C2—C3—C4	120.69 (15)	C2—C11—H11B	109.3
C2—C3—H3	119.7	C12—C11—H11B	109.3
C4—C3—H3	119.7	H11A—C11—H11B	108.0
C9—C4—C5	119.53 (14)	C13—C12—C17	118.60 (15)
C9—C4—C3	117.37 (15)	C13—C12—C11	121.08 (15)
C5—C4—C3	123.10 (15)	C17—C12—C11	120.30 (15)
C6—C5—C4	118.88 (15)	C14—C13—C12	120.44 (16)
C6—C5—H5	120.6	C14—C13—H13	119.8
C4—C5—H5	120.6	C12—C13—H13	119.8

C5—C6—C7	122.42 (15)	C15—C14—C13	120.45 (16)
C5—C6—Br1	119.21 (13)	C15—C14—H14	119.8
C7—C6—Br1	118.36 (12)	C13—C14—H14	119.8
C8—C7—C6	119.15 (16)	C14—C15—C16	119.61 (17)
C8—C7—H7	120.4	C14—C15—H15	120.2
C6—C7—H7	120.4	C16—C15—H15	120.2
C7—C8—C9	120.69 (16)	C15—C16—C17	120.23 (17)
C7—C8—H8	119.7	C15—C16—H16	119.9
C9—C8—H8	119.7	C17—C16—H16	119.9
N1—C9—C8	118.41 (15)	C16—C17—C12	120.66 (16)
N1—C9—C4	122.27 (14)	C16—C17—H17	119.7
C8—C9—C4	119.32 (15)	C12—C17—H17	119.7
O1—C10—H10A	109.5		

C9—N1—C1—O1	179.86 (14)	C1—N1—C9—C4	1.0 (2)
C9—N1—C1—C2	0.5 (2)	C7—C8—C9—N1	−179.29 (15)
C10—O1—C1—N1	2.2 (2)	C7—C8—C9—C4	1.0 (2)
C10—O1—C1—C2	−178.34 (16)	C5—C4—C9—N1	179.02 (14)
N1—C1—C2—C3	−2.0 (2)	C3—C4—C9—N1	−1.0 (2)
O1—C1—C2—C3	178.66 (14)	C5—C4—C9—C8	−1.2 (2)
N1—C1—C2—C11	175.74 (16)	C3—C4—C9—C8	178.69 (14)
O1—C1—C2—C11	−3.6 (2)	C3—C2—C11—C12	88.62 (19)
C1—C2—C3—C4	1.9 (2)	C1—C2—C11—C12	−88.96 (19)
C11—C2—C3—C4	−175.82 (14)	C2—C11—C12—C13	87.29 (18)
C2—C3—C4—C9	−0.5 (2)	C2—C11—C12—C17	−91.38 (18)
C2—C3—C4—C5	179.43 (14)	C17—C12—C13—C14	−0.1 (2)
C9—C4—C5—C6	1.0 (2)	C11—C12—C13—C14	−178.79 (15)
C3—C4—C5—C6	−178.96 (15)	C12—C13—C14—C15	−0.6 (3)
C4—C5—C6—C7	−0.4 (3)	C13—C14—C15—C16	0.6 (3)
C4—C5—C6—Br1	178.51 (11)	C14—C15—C16—C17	0.1 (3)
C5—C6—C7—C8	0.1 (3)	C15—C16—C17—C12	−0.7 (3)
Br1—C6—C7—C8	−178.82 (13)	C13—C12—C17—C16	0.7 (2)
C6—C7—C8—C9	−0.4 (2)	C11—C12—C17—C16	179.44 (15)
C1—N1—C9—C8	−178.70 (15)		

[4-(6-Bromo-2-methoxyquinolin-3-yl)-3-hydroxy-3-(naphthalen-1-yl)-4-phenylbutyl]dimethylazanium benzoate hydrate (4a)

Crystal data

$C_{32}H_{32}BrN_2O_2^+ \cdot C_7H_5O_2^- \cdot 1.166H_2O$
$M_r = 698.70$
Monoclinic, $P2_1$
$a = 12.6384$ (5) Å
$b = 7.9259$ (3) Å
$c = 17.5249$ (8) Å
$\beta = 99.8450$ (17)°
$V = 1729.63$ (12) Å^3
$Z = 2$
$F(000) = 727$
$D_x = 1.342$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9883 reflections
$\theta = 2.4$–31.0°
$\mu = 1.24$ mm^{-1}
$T = 150$ K
Rod, colourless
0.55 × 0.21 × 0.13 mm

Data collection

Bruker D8 Quest diffractometer with PhotonII charge-integrating pixel array detector (CPAD)
Radiation source: fine focus sealed tube X-ray source
Triumph curved graphite crystal monochromator
Detector resolution: 7.4074 pixels mm^{-1}
ω and phi scans
Absorption correction: multi-scan (SADABS2016; Krause *et al.*, 2015)
T_{min} = 0.638, T_{max} = 0.746
80228 measured reflections
13080 independent reflections
10456 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 33.2°, θ_{min} = 2.4°
h = −19→19
k = −11→12
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.073
S = 1.02
13080 reflections
445 parameters
5 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0275P)^2 + 0.0775P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}
Absolute structure: Flack x determined using 4051 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: 0.006 (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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Br1	1.08589 (2)	0.23491 (3)	−0.10932 (2)	0.03179 (6)	
O1	0.75158 (11)	0.32496 (17)	0.21175 (8)	0.0226 (3)	
H1B	0.726 (2)	0.2891 (12)	0.1673 (15)	0.034*	
O2	1.09216 (11)	0.03313 (19)	0.34490 (8)	0.0250 (3)	
O3	0.45306 (14)	0.2726 (2)	0.05187 (10)	0.0416 (4)	
O4	0.33269 (13)	0.4451 (2)	−0.01531 (8)	0.0313 (3)	
O5	0.67059 (14)	0.2732 (2)	0.06194 (9)	0.0333 (4)	
H5D	0.678 (3)	0.173 (5)	0.0399 (18)	0.050*	
H5E	0.609 (3)	0.282 (4)	0.0580 (18)	0.050*	
O6	1.3652 (11)	−0.1662 (18)	0.2755 (8)	0.061 (5)	0.166 (7)
H6D	1.381796	−0.060138	0.281851	0.091*	0.166 (7)
H6E	1.295716	−0.160377	0.260652	0.091*	0.166 (7)
N1	0.59081 (12)	−0.1394 (2)	0.14129 (9)	0.0209 (3)	
H1	0.621515	−0.107941	0.094301	0.025*	
N2	1.14361 (13)	0.0185 (2)	0.22591 (10)	0.0245 (3)	
C1	0.89931 (14)	0.1917 (2)	0.29663 (10)	0.0177 (3)	

H1A	0.912833	0.105727	0.338929	0.021*
C2	0.77558 (14)	0.1863 (2)	0.26328 (10)	0.0183 (3)
C3	0.74525 (14)	0.0197 (2)	0.21876 (10)	0.0189 (3)
H3A	0.773132	0.022355	0.169312	0.023*
H3B	0.779760	−0.075971	0.249780	0.023*
C4	0.62435 (14)	−0.0081 (3)	0.20190 (10)	0.0211 (3)
H4A	0.599796	−0.042486	0.250303	0.025*
H4B	0.588573	0.099757	0.184672	0.025*
C5	0.62902 (18)	−0.3115 (3)	0.16528 (14)	0.0318 (5)
H5A	0.607261	−0.390295	0.122359	0.048*
H5B	0.597537	−0.346948	0.210080	0.048*
H5C	0.707487	−0.311025	0.179237	0.048*
C6	0.47158 (16)	−0.1387 (3)	0.11978 (13)	0.0306 (4)
H6A	0.447484	−0.027521	0.099337	0.046*
H6B	0.439568	−0.163370	0.165684	0.046*
H6C	0.449243	−0.224808	0.080161	0.046*
C7	0.71486 (13)	0.2170 (3)	0.33194 (9)	0.0202 (3)
C8	0.66495 (17)	0.3697 (3)	0.33479 (12)	0.0292 (4)
H8	0.666506	0.447440	0.293730	0.035*
C9	0.6113 (2)	0.4171 (3)	0.39581 (13)	0.0376 (5)
H9	0.578160	0.524760	0.395368	0.045*
C10	0.6071 (2)	0.3088 (3)	0.45499 (13)	0.0365 (5)
H10	0.571006	0.340816	0.496032	0.044*
C11	0.65593 (18)	0.1492 (3)	0.45603 (12)	0.0315 (5)
C12	0.6518 (2)	0.0367 (4)	0.51821 (15)	0.0478 (7)
H12	0.615365	0.070222	0.558867	0.057*
C13	0.6981 (3)	−0.1164 (4)	0.52119 (18)	0.0583 (8)
H13	0.693912	−0.189679	0.563465	0.070*
C14	0.7528 (2)	−0.1682 (4)	0.46177 (16)	0.0469 (7)
H14	0.785004	−0.276756	0.463796	0.056*
C15	0.75993 (18)	−0.0622 (3)	0.40071 (12)	0.0304 (4)
H15	0.798548	−0.098306	0.361670	0.036*
C16	0.71136 (16)	0.0992 (3)	0.39441 (11)	0.0239 (4)
C17	0.93134 (14)	0.3615 (2)	0.33346 (10)	0.0187 (3)
C18	0.93961 (18)	0.3834 (3)	0.41341 (11)	0.0268 (4)
H18	0.925381	0.291144	0.444733	0.032*
C19	0.9683 (2)	0.5381 (3)	0.44745 (11)	0.0327 (5)
H19	0.973412	0.551193	0.501838	0.039*
C20	0.98963 (19)	0.6738 (3)	0.40278 (11)	0.0314 (5)
H20	1.010481	0.779369	0.426327	0.038*
C21	0.98022 (19)	0.6541 (3)	0.32309 (11)	0.0292 (4)
H21	0.993225	0.747126	0.291782	0.035*
C22	0.95196 (17)	0.4994 (3)	0.28939 (11)	0.0254 (4)
H22	0.946527	0.487081	0.234939	0.031*
C23	0.97303 (14)	0.1486 (2)	0.23939 (10)	0.0188 (3)
C24	0.95734 (14)	0.1960 (2)	0.16317 (10)	0.0199 (4)
H24	0.894360	0.256618	0.141861	0.024*
C25	1.03406 (14)	0.1555 (3)	0.11563 (11)	0.0209 (3)

C26	1.02197 (14)	0.2073 (3)	0.03709 (10)	0.0230 (4)
H26	0.961896	0.272839	0.014304	0.028*
C27	1.09881 (16)	0.1605 (3)	−0.00506 (11)	0.0239 (4)
C28	1.18724 (16)	0.0604 (3)	0.02554 (12)	0.0282 (4)
H28	1.237701	0.026726	−0.005945	0.034*
C29	1.19988 (17)	0.0116 (3)	0.10170 (13)	0.0289 (4)
H29	1.259621	−0.056265	0.123021	0.035*
C30	1.12503 (15)	0.0612 (3)	0.14873 (11)	0.0226 (4)
C31	1.07295 (15)	0.0640 (2)	0.26745 (11)	0.0207 (3)
C32	1.19411 (18)	−0.0419 (3)	0.37533 (14)	0.0364 (5)
H32A	1.197179	−0.156057	0.354303	0.055*
H32B	1.202846	−0.047604	0.431923	0.055*
H32C	1.251863	0.026661	0.360523	0.055*
C33	0.37747 (16)	0.3748 (3)	0.04720 (12)	0.0258 (4)
C34	0.33351 (16)	0.4182 (3)	0.11951 (11)	0.0244 (4)
C35	0.23897 (19)	0.5090 (3)	0.11598 (13)	0.0331 (5)
H35	0.202051	0.548308	0.067447	0.040*
C36	0.1981 (2)	0.5426 (4)	0.18289 (15)	0.0487 (7)
H36	0.133006	0.603990	0.179969	0.058*
C37	0.2515 (2)	0.4876 (5)	0.25356 (15)	0.0531 (8)
H37	0.223153	0.510905	0.299262	0.064*
C38	0.3462 (2)	0.3986 (4)	0.25814 (14)	0.0523 (8)
H38	0.383205	0.360736	0.306920	0.063*
C39	0.3871 (2)	0.3647 (4)	0.19136 (13)	0.0394 (6)
H39	0.452599	0.304104	0.194674	0.047*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03261 (10)	0.04237 (12)	0.02255 (8)	−0.00372 (11)	0.01087 (6)	−0.00332 (10)
O1	0.0289 (7)	0.0205 (7)	0.0173 (6)	0.0052 (5)	0.0002 (5)	0.0021 (5)
O2	0.0232 (6)	0.0262 (7)	0.0241 (6)	0.0044 (5)	−0.0002 (5)	0.0040 (6)
O3	0.0426 (9)	0.0432 (12)	0.0433 (9)	0.0160 (8)	0.0199 (7)	0.0064 (7)
O4	0.0368 (8)	0.0339 (8)	0.0242 (7)	0.0011 (7)	0.0077 (6)	0.0028 (6)
O5	0.0398 (8)	0.0321 (10)	0.0241 (7)	−0.0038 (7)	−0.0054 (6)	0.0037 (6)
O6	0.062 (9)	0.057 (9)	0.070 (10)	0.003 (6)	0.029 (7)	−0.006 (7)
N1	0.0207 (7)	0.0233 (8)	0.0181 (7)	−0.0019 (6)	0.0012 (6)	−0.0003 (6)
N2	0.0190 (7)	0.0245 (9)	0.0302 (8)	0.0031 (6)	0.0047 (6)	0.0034 (7)
C1	0.0204 (7)	0.0174 (9)	0.0154 (7)	0.0014 (6)	0.0036 (6)	0.0016 (6)
C2	0.0196 (7)	0.0195 (9)	0.0159 (7)	0.0018 (6)	0.0032 (6)	−0.0006 (6)
C3	0.0189 (8)	0.0194 (9)	0.0186 (7)	0.0017 (6)	0.0035 (6)	−0.0004 (6)
C4	0.0192 (8)	0.0245 (9)	0.0199 (8)	0.0000 (7)	0.0044 (6)	−0.0035 (7)
C5	0.0311 (10)	0.0230 (11)	0.0404 (11)	−0.0007 (8)	0.0036 (9)	0.0005 (8)
C6	0.0209 (9)	0.0387 (12)	0.0301 (10)	−0.0047 (8)	−0.0022 (8)	0.0033 (9)
C7	0.0202 (7)	0.0236 (10)	0.0166 (6)	0.0026 (8)	0.0023 (5)	−0.0022 (7)
C8	0.0322 (10)	0.0302 (11)	0.0265 (9)	0.0085 (9)	0.0091 (8)	0.0004 (8)
C9	0.0412 (12)	0.0380 (13)	0.0367 (12)	0.0113 (10)	0.0158 (10)	−0.0068 (10)
C10	0.0343 (11)	0.0499 (14)	0.0285 (10)	0.0041 (10)	0.0144 (9)	−0.0078 (10)

C11	0.0292 (11)	0.0426 (13)	0.0251 (9)	−0.0040 (10)	0.0112 (8)	−0.0013 (9)
C12	0.0601 (17)	0.0552 (17)	0.0344 (12)	0.0006 (14)	0.0257 (12)	0.0081 (12)
C13	0.080 (2)	0.0565 (19)	0.0468 (16)	0.0065 (16)	0.0342 (15)	0.0234 (14)
C14	0.0606 (17)	0.0397 (15)	0.0450 (14)	0.0093 (12)	0.0225 (12)	0.0186 (12)
C15	0.0355 (11)	0.0291 (11)	0.0284 (10)	0.0000 (9)	0.0105 (8)	0.0047 (8)
C16	0.0234 (9)	0.0281 (10)	0.0205 (8)	−0.0027 (7)	0.0050 (7)	−0.0008 (7)
C17	0.0206 (8)	0.0189 (8)	0.0159 (7)	0.0014 (7)	0.0008 (6)	0.0010 (6)
C18	0.0401 (11)	0.0225 (10)	0.0165 (8)	−0.0047 (8)	0.0008 (7)	0.0027 (7)
C19	0.0522 (13)	0.0290 (11)	0.0154 (8)	−0.0069 (10)	0.0018 (8)	−0.0016 (8)
C20	0.0500 (13)	0.0219 (10)	0.0209 (9)	−0.0066 (9)	0.0020 (8)	−0.0027 (8)
C21	0.0471 (12)	0.0200 (10)	0.0206 (8)	−0.0058 (9)	0.0058 (8)	0.0013 (7)
C22	0.0379 (11)	0.0223 (10)	0.0169 (8)	−0.0046 (8)	0.0068 (7)	0.0003 (7)
C23	0.0179 (8)	0.0164 (9)	0.0220 (8)	0.0001 (6)	0.0032 (6)	−0.0011 (7)
C24	0.0184 (7)	0.0212 (10)	0.0204 (7)	0.0026 (6)	0.0042 (6)	0.0005 (6)
C25	0.0188 (8)	0.0212 (9)	0.0238 (8)	−0.0004 (7)	0.0063 (6)	−0.0015 (7)
C26	0.0221 (8)	0.0252 (11)	0.0223 (7)	0.0006 (7)	0.0054 (6)	−0.0013 (7)
C27	0.0259 (9)	0.0238 (9)	0.0237 (8)	−0.0052 (7)	0.0093 (7)	−0.0043 (8)
C28	0.0222 (9)	0.0302 (11)	0.0349 (11)	−0.0013 (8)	0.0125 (8)	−0.0059 (9)
C29	0.0212 (9)	0.0293 (11)	0.0378 (11)	0.0035 (8)	0.0100 (8)	0.0003 (9)
C30	0.0195 (8)	0.0209 (9)	0.0283 (9)	0.0004 (7)	0.0062 (7)	0.0001 (7)
C31	0.0200 (8)	0.0174 (8)	0.0238 (8)	−0.0014 (7)	0.0013 (7)	0.0011 (7)
C32	0.0299 (11)	0.0417 (14)	0.0333 (11)	0.0097 (10)	−0.0070 (9)	0.0046 (10)
C33	0.0273 (9)	0.0243 (10)	0.0272 (9)	−0.0023 (8)	0.0083 (7)	0.0008 (8)
C34	0.0254 (9)	0.0241 (10)	0.0240 (9)	0.0001 (7)	0.0049 (7)	−0.0013 (7)
C35	0.0313 (11)	0.0417 (14)	0.0259 (10)	0.0102 (10)	0.0042 (8)	0.0009 (9)
C36	0.0409 (13)	0.0683 (19)	0.0383 (13)	0.0234 (13)	0.0110 (11)	−0.0028 (13)
C37	0.0581 (17)	0.076 (2)	0.0279 (11)	0.0201 (15)	0.0141 (11)	−0.0082 (13)
C38	0.0554 (16)	0.076 (2)	0.0248 (11)	0.0252 (15)	0.0053 (11)	0.0020 (12)
C39	0.0383 (12)	0.0507 (15)	0.0294 (11)	0.0190 (11)	0.0061 (9)	0.0023 (10)

Geometric parameters (Å, º)

Br1—C27	1.900 (2)	C13—C14	1.406 (4)
O1—C2	1.422 (2)	C13—H13	0.9500
O1—H1B	0.84 (3)	C14—C15	1.375 (3)
O2—C31	1.360 (2)	C14—H14	0.9500
O2—C32	1.437 (2)	C15—C16	1.414 (3)
O3—C33	1.244 (3)	C15—H15	0.9500
O4—C33	1.273 (3)	C17—C22	1.388 (3)
O5—H5D	0.90 (4)	C17—C18	1.398 (3)
O5—H5E	0.77 (3)	C18—C19	1.385 (3)
O6—H6D	0.8692	C18—H18	0.9500
O6—H6E	0.8735	C19—C20	1.383 (3)
N1—C5	1.484 (3)	C19—H19	0.9500
N1—C6	1.489 (2)	C20—C21	1.390 (3)
N1—C4	1.495 (2)	C20—H20	0.9500
N1—H1	1.0000	C21—C22	1.381 (3)
N2—C31	1.296 (2)	C21—H21	0.9500

N2—C30	1.375 (3)	C22—H22	0.9500
C1—C17	1.517 (2)	C23—C24	1.369 (2)
C1—C23	1.520 (2)	C23—C31	1.440 (3)
C1—C2	1.574 (2)	C24—C25	1.419 (2)
C1—H1A	1.0000	C24—H24	0.9500
C2—C3	1.548 (3)	C25—C30	1.410 (3)
C2—C7	1.553 (2)	C25—C26	1.419 (3)
C3—C4	1.522 (2)	C26—C27	1.368 (3)
C3—H3A	0.9900	C26—H26	0.9500
C3—H3B	0.9900	C27—C28	1.401 (3)
C4—H4A	0.9900	C28—C29	1.372 (3)
C4—H4B	0.9900	C28—H28	0.9500
C5—H5A	0.9800	C29—C30	1.413 (3)
C5—H5B	0.9800	C29—H29	0.9500
C5—H5C	0.9800	C32—H32A	0.9800
C6—H6A	0.9800	C32—H32B	0.9800
C6—H6B	0.9800	C32—H32C	0.9800
C6—H6C	0.9800	C33—C34	1.508 (3)
C7—C8	1.369 (3)	C34—C35	1.387 (3)
C7—C16	1.446 (3)	C34—C39	1.390 (3)
C8—C9	1.412 (3)	C35—C36	1.385 (3)
C8—H8	0.9500	C35—H35	0.9500
C9—C10	1.354 (4)	C36—C37	1.376 (4)
C9—H9	0.9500	C36—H36	0.9500
C10—C11	1.406 (3)	C37—C38	1.380 (4)
C10—H10	0.9500	C37—H37	0.9500
C11—C12	1.416 (3)	C38—C39	1.384 (3)
C11—C16	1.440 (3)	C38—H38	0.9500
C12—C13	1.345 (5)	C39—H39	0.9500
C12—H12	0.9500		

C2—O1—H1B	109.5	C15—C16—C7	125.05 (18)
C31—O2—C32	116.16 (16)	C11—C16—C7	118.14 (19)
H5D—O5—H5E	103 (3)	C22—C17—C18	118.08 (17)
H6D—O6—H6E	101.2	C22—C17—C1	121.56 (15)
C5—N1—C6	109.94 (17)	C18—C17—C1	120.36 (16)
C5—N1—C4	113.54 (15)	C19—C18—C17	120.74 (18)
C6—N1—C4	109.21 (16)	C19—C18—H18	119.6
C5—N1—H1	108.0	C17—C18—H18	119.6
C6—N1—H1	108.0	C20—C19—C18	120.44 (18)
C4—N1—H1	108.0	C20—C19—H19	119.8
C31—N2—C30	117.76 (16)	C18—C19—H19	119.8
C17—C1—C23	109.33 (14)	C19—C20—C21	119.3 (2)
C17—C1—C2	110.99 (14)	C19—C20—H20	120.3
C23—C1—C2	115.54 (14)	C21—C20—H20	120.3
C17—C1—H1A	106.8	C22—C21—C20	120.05 (19)
C23—C1—H1A	106.8	C22—C21—H21	120.0
C2—C1—H1A	106.8	C20—C21—H21	120.0

O1—C2—C3	109.36 (14)	C21—C22—C17	121.36 (17)
O1—C2—C7	106.90 (15)	C21—C22—H22	119.3
C3—C2—C7	114.32 (15)	C17—C22—H22	119.3
O1—C2—C1	107.77 (14)	C24—C23—C31	115.67 (16)
C3—C2—C1	110.81 (14)	C24—C23—C1	125.41 (16)
C7—C2—C1	107.42 (13)	C31—C23—C1	118.68 (16)
C4—C3—C2	112.06 (15)	C23—C24—C25	120.77 (16)
C4—C3—H3A	109.2	C23—C24—H24	119.6
C2—C3—H3A	109.2	C25—C24—H24	119.6
C4—C3—H3B	109.2	C30—C25—C24	117.98 (17)
C2—C3—H3B	109.2	C30—C25—C26	119.80 (16)
H3A—C3—H3B	107.9	C24—C25—C26	122.23 (17)
N1—C4—C3	113.09 (15)	C27—C26—C25	118.48 (18)
N1—C4—H4A	109.0	C27—C26—H26	120.8
C3—C4—H4A	109.0	C25—C26—H26	120.8
N1—C4—H4B	109.0	C26—C27—C28	122.72 (19)
C3—C4—H4B	109.0	C26—C27—Br1	118.87 (16)
H4A—C4—H4B	107.8	C28—C27—Br1	118.41 (14)
N1—C5—H5A	109.5	C29—C28—C27	118.98 (18)
N1—C5—H5B	109.5	C29—C28—H28	120.5
H5A—C5—H5B	109.5	C27—C28—H28	120.5
N1—C5—H5C	109.5	C28—C29—C30	120.7 (2)
H5A—C5—H5C	109.5	C28—C29—H29	119.6
H5B—C5—H5C	109.5	C30—C29—H29	119.6
N1—C6—H6A	109.5	N2—C30—C25	121.68 (17)
N1—C6—H6B	109.5	N2—C30—C29	119.12 (18)
H6A—C6—H6B	109.5	C25—C30—C29	119.19 (18)
N1—C6—H6C	109.5	N2—C31—O2	119.21 (17)
H6A—C6—H6C	109.5	N2—C31—C23	125.87 (17)
H6B—C6—H6C	109.5	O2—C31—C23	114.92 (16)
C8—C7—C16	118.01 (16)	O2—C32—H32A	109.5
C8—C7—C2	117.30 (17)	O2—C32—H32B	109.5
C16—C7—C2	124.64 (18)	H32A—C32—H32B	109.5
C7—C8—C9	123.1 (2)	O2—C32—H32C	109.5
C7—C8—H8	118.4	H32A—C32—H32C	109.5
C9—C8—H8	118.4	H32B—C32—H32C	109.5
C10—C9—C8	119.9 (2)	O3—C33—O4	124.42 (19)
C10—C9—H9	120.1	O3—C33—C34	118.83 (19)
C8—C9—H9	120.1	O4—C33—C34	116.74 (18)
C9—C10—C11	120.4 (2)	C35—C34—C39	118.76 (19)
C9—C10—H10	119.8	C35—C34—C33	121.28 (18)
C11—C10—H10	119.8	C39—C34—C33	119.95 (19)
C10—C11—C12	120.2 (2)	C36—C35—C34	120.3 (2)
C10—C11—C16	120.5 (2)	C36—C35—H35	119.8
C12—C11—C16	119.3 (2)	C34—C35—H35	119.8
C13—C12—C11	121.7 (2)	C37—C36—C35	120.3 (2)
C13—C12—H12	119.2	C37—C36—H36	119.9
C11—C12—H12	119.2	C35—C36—H36	119.9

C12—C13—C14	120.0 (2)	C36—C37—C38	120.1 (2)
C12—C13—H13	120.0	C36—C37—H37	120.0
C14—C13—H13	120.0	C38—C37—H37	120.0
C15—C14—C13	120.3 (3)	C37—C38—C39	119.7 (2)
C15—C14—H14	119.9	C37—C38—H38	120.1
C13—C14—H14	119.9	C39—C38—H38	120.1
C14—C15—C16	121.9 (2)	C38—C39—C34	120.8 (2)
C14—C15—H15	119.1	C38—C39—H39	119.6
C16—C15—H15	119.1	C34—C39—H39	119.6
C15—C16—C11	116.81 (19)		

C17—C1—C2—O1	54.82 (17)	C19—C20—C21—C22	−1.3 (4)
C23—C1—C2—O1	−70.37 (18)	C20—C21—C22—C17	0.7 (3)
C17—C1—C2—C3	174.44 (14)	C18—C17—C22—C21	0.2 (3)
C23—C1—C2—C3	49.3 (2)	C1—C17—C22—C21	179.44 (19)
C17—C1—C2—C7	−60.05 (19)	C17—C1—C23—C24	−87.3 (2)
C23—C1—C2—C7	174.77 (16)	C2—C1—C23—C24	38.7 (2)
O1—C2—C3—C4	−74.49 (18)	C17—C1—C23—C31	86.83 (19)
C7—C2—C3—C4	45.3 (2)	C2—C1—C23—C31	−147.14 (16)
C1—C2—C3—C4	166.84 (14)	C31—C23—C24—C25	2.7 (3)
C5—N1—C4—C3	65.8 (2)	C1—C23—C24—C25	177.00 (17)
C6—N1—C4—C3	−171.11 (16)	C23—C24—C25—C30	1.8 (3)
C2—C3—C4—N1	164.04 (15)	C23—C24—C25—C26	−177.98 (18)
O1—C2—C7—C8	−7.6 (2)	C30—C25—C26—C27	1.3 (3)
C3—C2—C7—C8	−128.77 (19)	C24—C25—C26—C27	−178.92 (18)
C1—C2—C7—C8	107.84 (18)	C25—C26—C27—C28	1.7 (3)
O1—C2—C7—C16	175.18 (17)	C25—C26—C27—Br1	−178.04 (14)
C3—C2—C7—C16	54.0 (2)	C26—C27—C28—C29	−2.4 (3)
C1—C2—C7—C16	−69.4 (2)	Br1—C27—C28—C29	177.34 (17)
C16—C7—C8—C9	0.4 (3)	C27—C28—C29—C30	0.0 (3)
C2—C7—C8—C9	−177.0 (2)	C31—N2—C30—C25	1.8 (3)
C7—C8—C9—C10	−0.4 (4)	C31—N2—C30—C29	−179.23 (19)
C8—C9—C10—C11	0.0 (4)	C24—C25—C30—N2	−4.3 (3)
C9—C10—C11—C12	179.7 (3)	C26—C25—C30—N2	175.47 (18)
C9—C10—C11—C16	0.3 (3)	C24—C25—C30—C29	176.72 (18)
C10—C11—C12—C13	−179.6 (3)	C26—C25—C30—C29	−3.5 (3)
C16—C11—C12—C13	−0.2 (4)	C28—C29—C30—N2	−176.2 (2)
C11—C12—C13—C14	0.3 (5)	C28—C29—C30—C25	2.8 (3)
C12—C13—C14—C15	0.5 (5)	C30—N2—C31—O2	−175.89 (17)
C13—C14—C15—C16	−1.4 (4)	C30—N2—C31—C23	3.4 (3)
C14—C15—C16—C11	1.4 (3)	C32—O2—C31—N2	3.0 (3)
C14—C15—C16—C7	−179.7 (2)	C32—O2—C31—C23	−176.38 (18)
C10—C11—C16—C15	178.8 (2)	C24—C23—C31—N2	−5.7 (3)
C12—C11—C16—C15	−0.6 (3)	C1—C23—C31—N2	179.65 (18)
C10—C11—C16—C7	−0.2 (3)	C24—C23—C31—O2	173.63 (16)
C12—C11—C16—C7	−179.6 (2)	C1—C23—C31—O2	−1.1 (2)
C8—C7—C16—C15	−179.1 (2)	O3—C33—C34—C35	−169.4 (2)
C2—C7—C16—C15	−1.9 (3)	O4—C33—C34—C35	9.7 (3)

C8—C7—C16—C11	−0.2 (3)	O3—C33—C34—C39	9.6 (3)
C2—C7—C16—C11	177.02 (17)	O4—C33—C34—C39	−171.3 (2)
C23—C1—C17—C22	46.1 (2)	C39—C34—C35—C36	−1.2 (4)
C2—C1—C17—C22	−82.5 (2)	C33—C34—C35—C36	177.8 (3)
C23—C1—C17—C18	−134.71 (18)	C34—C35—C36—C37	0.6 (5)
C2—C1—C17—C18	96.7 (2)	C35—C36—C37—C38	0.1 (5)
C22—C17—C18—C19	−0.5 (3)	C36—C37—C38—C39	−0.1 (5)
C1—C17—C18—C19	−179.7 (2)	C37—C38—C39—C34	−0.5 (5)
C17—C18—C19—C20	−0.2 (4)	C35—C34—C39—C38	1.1 (4)
C18—C19—C20—C21	1.1 (4)	C33—C34—C39—C38	−177.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O5	0.84	1.87	2.681 (2)	164
O5—H5*D*···O4^{i}	0.90 (4)	1.85 (4)	2.724 (2)	163 (3)
O5—H5*E*···O3	0.77 (3)	1.96 (3)	2.724 (2)	172 (4)
O6—H6*E*···N2	0.87	2.38	3.148 (13)	147
N1—H1···O4^{i}	1.00	1.64	2.643 (2)	178
C5—H5*C*···Br1^{ii}	0.98	3.09	3.910 (2)	142
C6—H6*A*···O3	0.98	2.53	3.465 (3)	161
C6—H6*B*···O6^{iii}	0.98	2.28	3.249 (13)	169
C28—H28···O5^{ii}	0.95	2.59	3.421 (3)	146

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

[4-(6-Bromo-2-methoxyquinolin-3-yl)-3-hydroxy-3-(naphthalen-1-yl)-4-phenylbutyl]dimethylazanium benzoate acetonitrile 0.742-solvate monohydrate (4b)

Crystal data

$C_{32}H_{32}BrN_2O_2^+ \cdot C_7H_5O_2^- \cdot 0.742C_2H_3N \cdot H_2O$
M_r = 726.10
Monoclinic, $P2_1$
a = 12.8661 (8) Å
b = 8.0386 (5) Å
c = 17.4704 (10) Å
β = 101.093 (3)°
V = 1773.13 (19) Å^3
Z = 2
$F(000)$ = 757
D_x = 1.360 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9023 reflections
θ = 2.6–79.1°
μ = 1.97 mm^{-1}
T = 150 K
Needle, colourless
0.31 × 0.05 × 0.05 mm

Data collection

Bruker D8 Quest diffractometer with PhotonIII_C14 charge-integrating and photon counting pixel array detector
Radiation source: I-mu-S microsource X-ray tube
Laterally graded multilayer (Goebel) mirror monochromator
Detector resolution: 7.4074 pixels mm^{-1}
ω and phi scans
Absorption correction: multi-scan (SADABS2016; Krause *et al.*, 2015)
T_{min} = 0.599, T_{max} = 0.754
39739 measured reflections
7360 independent reflections
6750 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 80.1°, θ_{min} = 2.6°
h = −16→16
k = −8→10
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.085$
$S = 1.06$
7360 reflections
515 parameters
195 restraints
Primary atom site location: isomorphous structure methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0233P)^2 + 1.042P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ e Å^{-3}
Absolute structure: Flack *x* determined using 2778 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: 0.004 (8)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Br1	1.09186 (3)	0.23149 (6)	−0.10436 (2)	0.04184 (12)	
O1	0.7542 (2)	0.3259 (3)	0.20992 (15)	0.0271 (5)	
H1B	0.734 (4)	0.292 (2)	0.168 (3)	0.041*	
O2	1.08646 (19)	0.0320 (3)	0.35039 (14)	0.0297 (5)	
O3	0.4583 (2)	0.2868 (4)	0.05847 (18)	0.0497 (8)	
O4	0.3385 (2)	0.4448 (4)	−0.01724 (15)	0.0373 (6)	
O5	0.6707 (3)	0.2738 (4)	0.05844 (16)	0.0387 (7)	
H5D	0.674 (5)	0.174 (4)	0.041 (3)	0.058*	
H5E	0.607 (2)	0.288 (7)	0.058 (3)	0.058*	
N1	0.5911 (2)	−0.1342 (4)	0.14353 (17)	0.0272 (6)	
H1	0.618783	−0.106871	0.095314	0.033*	
N2	1.1414 (2)	0.0196 (4)	0.23270 (18)	0.0292 (6)	
C1	0.9000 (2)	0.1962 (4)	0.29767 (17)	0.0228 (7)	
H1A	0.913127	0.113403	0.341185	0.027*	
C2	0.7781 (3)	0.1907 (4)	0.26263 (18)	0.0228 (7)	
C3	0.7471 (3)	0.0265 (4)	0.21840 (19)	0.0234 (7)	
H3A	0.772164	0.028938	0.168253	0.028*	
H3B	0.782695	−0.067448	0.249584	0.028*	
C4	0.6277 (3)	−0.0017 (5)	0.20251 (19)	0.0249 (7)	
H4A	0.605984	−0.032128	0.252035	0.030*	
H4B	0.591887	0.103914	0.184090	0.030*	
C5	0.6308 (3)	−0.3018 (5)	0.1703 (2)	0.0391 (9)	
H5A	0.605300	−0.383658	0.129406	0.059*	
H5B	0.604938	−0.331264	0.217728	0.059*	
H5C	0.708438	−0.301159	0.181360	0.059*	
C6	0.4732 (3)	−0.1344 (6)	0.1230 (2)	0.0366 (9)	
H6A	0.448325	−0.025168	0.102189	0.055*	

H6B	0.444151	−0.158068	0.169733	0.055*
H6C	0.449601	−0.219985	0.083535	0.055*
C7	0.7184 (2)	0.2225 (5)	0.33036 (16)	0.0237 (6)
C8	0.6696 (3)	0.3737 (5)	0.3321 (2)	0.0322 (8)
H8	0.670541	0.448836	0.290297	0.039*
C9	0.6182 (3)	0.4236 (6)	0.3928 (2)	0.0391 (9)
H9	0.586471	0.530466	0.391630	0.047*
C10	0.6143 (3)	0.3178 (6)	0.4529 (2)	0.0371 (9)
H10	0.578544	0.350206	0.493248	0.045*
C11	0.6629 (3)	0.1604 (5)	0.4556 (2)	0.0328 (8)
C12	0.6601 (4)	0.0520 (6)	0.5190 (2)	0.0464 (11)
H12	0.624115	0.086173	0.559045	0.056*
C13	0.7070 (5)	−0.0985 (7)	0.5241 (3)	0.0578 (14)
H13	0.704267	−0.168798	0.567337	0.069*
C14	0.7601 (4)	−0.1511 (6)	0.4650 (3)	0.0477 (11)
H14	0.793019	−0.257285	0.468543	0.057*
C15	0.7646 (3)	−0.0505 (5)	0.4024 (2)	0.0329 (8)
H15	0.800873	−0.088643	0.363250	0.040*
C16	0.7167 (3)	0.1088 (5)	0.3945 (2)	0.0261 (7)
C17	0.9317 (3)	0.3644 (4)	0.33358 (19)	0.0243 (7)
C18	0.9390 (3)	0.3895 (5)	0.41323 (19)	0.0287 (7)
H18	0.925588	0.299474	0.445188	0.034*
C19	0.9654 (3)	0.5436 (5)	0.4466 (2)	0.0319 (8)
H19	0.970429	0.558180	0.501130	0.038*
C20	0.9846 (3)	0.6768 (5)	0.4009 (2)	0.0340 (8)
H20	1.002007	0.782928	0.423684	0.041*
C21	0.9780 (3)	0.6530 (5)	0.3214 (2)	0.0341 (8)
H21	0.991414	0.743347	0.289646	0.041*
C22	0.9520 (3)	0.4987 (5)	0.2883 (2)	0.0301 (8)
H22	0.947839	0.484164	0.233828	0.036*
C23	0.9726 (3)	0.1487 (4)	0.2416 (2)	0.0241 (7)
C24	0.9580 (3)	0.1938 (4)	0.16468 (18)	0.0242 (7)
H24	0.896018	0.253246	0.141633	0.029*
C25	1.0347 (3)	0.1528 (5)	0.1189 (2)	0.0263 (7)
C26	1.0240 (3)	0.2018 (5)	0.04000 (19)	0.0289 (8)
H26	0.964322	0.264196	0.015139	0.035*
C27	1.1014 (3)	0.1574 (5)	0.0004 (2)	0.0312 (8)
C28	1.1884 (3)	0.0584 (5)	0.0338 (2)	0.0361 (9)
H28	1.238547	0.023743	0.003564	0.043*
C29	1.2000 (3)	0.0124 (6)	0.1104 (3)	0.0378 (9)
H29	1.259208	−0.052812	0.133734	0.045*
C30	1.1246 (3)	0.0612 (5)	0.1550 (2)	0.0285 (7)
C31	1.0703 (3)	0.0644 (4)	0.2727 (2)	0.0251 (7)
C32	1.1850 (3)	−0.0452 (6)	0.3846 (3)	0.0427 (10)
H32A	1.191281	−0.152129	0.358991	0.064*
H32B	1.187118	−0.063515	0.440374	0.064*
H32C	1.243771	0.027348	0.377926	0.064*
C33	0.3819 (3)	0.3840 (5)	0.0484 (2)	0.0310 (8)

C34	0.3376 (12)	0.439 (3)	0.1162 (6)	0.030 (2)	0.742 (7)
C35	0.2342 (10)	0.5018 (15)	0.1055 (5)	0.0338 (19)	0.742 (7)
H35	0.193862	0.513732	0.054183	0.041*	0.742 (7)
C36	0.1905 (9)	0.5461 (17)	0.1683 (5)	0.047 (2)	0.742 (7)
H36	0.118077	0.577683	0.160811	0.057*	0.742 (7)
C37	0.2530 (7)	0.5447 (12)	0.2437 (5)	0.0531 (19)	0.742 (7)
H37	0.225227	0.586384	0.286502	0.064*	0.742 (7)
C38	0.3550 (6)	0.4828 (10)	0.2555 (4)	0.0467 (16)	0.742 (7)
H38	0.396204	0.475671	0.306703	0.056*	0.742 (7)
C39	0.3966 (5)	0.4313 (9)	0.1920 (4)	0.0371 (14)	0.742 (7)
H39	0.466935	0.389697	0.200206	0.044*	0.742 (7)
C34B	0.331 (4)	0.429 (8)	0.1208 (16)	0.0242 (7)	0.258 (7)
C35B	0.239 (3)	0.526 (5)	0.1165 (17)	0.035 (4)	0.258 (7)
H35B	0.202750	0.564683	0.067136	0.042*	0.258 (7)
C36B	0.202 (3)	0.567 (5)	0.1825 (16)	0.044 (4)	0.258 (7)
H36B	0.150076	0.651410	0.181779	0.053*	0.258 (7)
C37B	0.2432 (18)	0.479 (3)	0.2514 (14)	0.047 (3)	0.258 (7)
H37B	0.205535	0.477535	0.293157	0.056*	0.258 (7)
C38B	0.3375 (16)	0.396 (3)	0.2586 (11)	0.047 (3)	0.258 (7)
H38B	0.373369	0.359002	0.308434	0.057*	0.258 (7)
C39B	0.3805 (17)	0.365 (3)	0.1932 (11)	0.039 (3)	0.258 (7)
H39B	0.443068	0.299799	0.197365	0.047*	0.258 (7)
N3	0.4791 (5)	−0.2008 (8)	0.3556 (4)	0.0620 (18)	0.742 (7)
C40	0.4502 (5)	−0.0703 (9)	0.3379 (4)	0.0477 (16)	0.742 (7)
C41	0.4178 (6)	0.0985 (10)	0.3164 (5)	0.063 (2)	0.742 (7)
H41A	0.473979	0.155009	0.295727	0.12 (4)*	0.742 (7)
H41B	0.404449	0.158210	0.362451	0.14 (5)*	0.742 (7)
H41C	0.352857	0.096518	0.276432	0.06 (2)*	0.742 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0403 (2)	0.0597 (3)	0.02848 (18)	−0.0089 (2)	0.01393 (14)	−0.0069 (2)
O1	0.0346 (14)	0.0229 (13)	0.0218 (12)	0.0020 (10)	0.0004 (10)	0.0026 (9)
O2	0.0255 (12)	0.0329 (14)	0.0287 (13)	0.0057 (10)	−0.0002 (10)	0.0040 (11)
O3	0.0501 (17)	0.053 (2)	0.0513 (17)	0.0218 (14)	0.0217 (14)	0.0106 (14)
O4	0.0429 (16)	0.0428 (17)	0.0267 (13)	0.0012 (13)	0.0080 (11)	0.0042 (12)
O5	0.0430 (15)	0.0396 (19)	0.0296 (14)	0.0001 (12)	−0.0030 (12)	0.0026 (11)
N1	0.0259 (15)	0.0300 (17)	0.0247 (14)	−0.0036 (12)	0.0020 (11)	−0.0002 (12)
N2	0.0236 (14)	0.0298 (17)	0.0348 (16)	0.0020 (12)	0.0075 (12)	−0.0010 (13)
C1	0.0249 (15)	0.025 (2)	0.0189 (14)	0.0024 (13)	0.0057 (11)	0.0019 (12)
C2	0.0264 (15)	0.0230 (19)	0.0186 (14)	0.0018 (12)	0.0036 (12)	0.0011 (12)
C3	0.0253 (16)	0.0233 (17)	0.0216 (15)	0.0032 (13)	0.0043 (12)	−0.0013 (13)
C4	0.0269 (17)	0.0279 (19)	0.0200 (16)	−0.0004 (14)	0.0049 (13)	−0.0038 (13)
C5	0.042 (2)	0.030 (2)	0.044 (2)	−0.0019 (17)	0.0062 (16)	0.0004 (17)
C6	0.0284 (19)	0.046 (2)	0.0318 (19)	−0.0067 (16)	−0.0032 (15)	0.0013 (17)
C7	0.0213 (13)	0.0262 (17)	0.0226 (13)	0.0029 (16)	0.0014 (10)	−0.0024 (16)
C8	0.0338 (19)	0.034 (2)	0.0292 (18)	0.0083 (16)	0.0055 (15)	0.0040 (15)

C9	0.042 (2)	0.037 (2)	0.038 (2)	0.0124 (18)	0.0085 (17)	−0.0050 (17)
C10	0.036 (2)	0.046 (2)	0.032 (2)	0.0070 (18)	0.0116 (17)	−0.0042 (17)
C11	0.0315 (19)	0.042 (2)	0.0249 (18)	0.0011 (16)	0.0068 (15)	0.0003 (15)
C12	0.056 (3)	0.054 (3)	0.035 (2)	0.006 (2)	0.025 (2)	0.007 (2)
C13	0.077 (3)	0.062 (3)	0.043 (3)	0.015 (3)	0.033 (2)	0.023 (2)
C14	0.064 (3)	0.043 (3)	0.041 (2)	0.011 (2)	0.023 (2)	0.0151 (19)
C15	0.037 (2)	0.031 (2)	0.0324 (19)	0.0024 (16)	0.0111 (16)	0.0040 (15)
C16	0.0240 (16)	0.0307 (19)	0.0239 (17)	0.0004 (13)	0.0052 (13)	0.0014 (13)
C17	0.0229 (16)	0.0269 (18)	0.0223 (16)	0.0006 (13)	0.0022 (12)	0.0014 (13)
C18	0.0379 (19)	0.0275 (19)	0.0197 (16)	−0.0008 (15)	0.0027 (14)	0.0023 (13)
C19	0.044 (2)	0.031 (2)	0.0197 (16)	−0.0037 (16)	0.0048 (15)	−0.0011 (14)
C20	0.048 (2)	0.027 (2)	0.0261 (17)	−0.0024 (15)	0.0028 (15)	−0.0029 (13)
C21	0.049 (2)	0.029 (2)	0.0240 (17)	−0.0065 (17)	0.0074 (15)	0.0036 (15)
C22	0.041 (2)	0.030 (2)	0.0204 (16)	−0.0047 (15)	0.0078 (14)	−0.0004 (14)
C23	0.0220 (16)	0.0228 (17)	0.0275 (17)	0.0007 (13)	0.0045 (13)	−0.0019 (13)
C24	0.0239 (14)	0.0259 (19)	0.0232 (14)	0.0014 (12)	0.0060 (12)	−0.0018 (12)
C25	0.0244 (16)	0.0262 (18)	0.0300 (18)	−0.0026 (13)	0.0091 (13)	−0.0038 (14)
C26	0.0284 (16)	0.034 (2)	0.0251 (15)	−0.0016 (14)	0.0087 (12)	−0.0039 (14)
C27	0.0326 (19)	0.034 (2)	0.0287 (18)	−0.0096 (16)	0.0104 (15)	−0.0061 (15)
C28	0.0308 (19)	0.039 (2)	0.043 (2)	−0.0012 (16)	0.0185 (16)	−0.0075 (17)
C29	0.0283 (19)	0.038 (2)	0.049 (2)	0.0038 (16)	0.0120 (17)	−0.0006 (18)
C30	0.0250 (17)	0.0272 (19)	0.0341 (19)	−0.0004 (14)	0.0074 (14)	−0.0022 (15)
C31	0.0253 (16)	0.0220 (17)	0.0270 (17)	−0.0010 (13)	0.0025 (13)	0.0024 (13)
C32	0.030 (2)	0.051 (3)	0.042 (2)	0.0104 (18)	−0.0054 (17)	0.0067 (19)
C33	0.0353 (19)	0.029 (2)	0.0299 (18)	−0.0005 (15)	0.0096 (15)	0.0002 (15)
C34	0.030 (3)	0.035 (4)	0.027 (3)	−0.001 (3)	0.006 (2)	−0.001 (2)
C35	0.034 (3)	0.035 (4)	0.032 (3)	0.004 (3)	0.006 (3)	−0.005 (3)
C36	0.041 (4)	0.060 (5)	0.042 (4)	0.012 (4)	0.010 (3)	−0.013 (4)
C37	0.065 (4)	0.058 (5)	0.040 (3)	0.010 (4)	0.019 (3)	−0.009 (3)
C38	0.056 (3)	0.053 (4)	0.030 (3)	0.002 (3)	0.005 (2)	−0.007 (3)
C39	0.036 (3)	0.040 (4)	0.034 (3)	0.000 (3)	0.003 (2)	−0.001 (3)
C34B	0.0239 (14)	0.0259 (19)	0.0232 (14)	0.0014 (12)	0.0060 (12)	−0.0018 (12)
C35B	0.031 (6)	0.042 (7)	0.030 (6)	0.003 (6)	0.003 (5)	−0.006 (6)
C36B	0.044 (6)	0.052 (7)	0.037 (6)	0.009 (6)	0.012 (6)	−0.017 (6)
C37B	0.053 (6)	0.055 (7)	0.036 (6)	0.003 (6)	0.018 (5)	−0.009 (6)
C38B	0.052 (6)	0.056 (7)	0.036 (5)	0.004 (6)	0.012 (5)	−0.003 (6)
C39B	0.040 (6)	0.044 (6)	0.033 (5)	0.004 (6)	0.008 (5)	−0.004 (6)
N3	0.077 (4)	0.038 (3)	0.078 (4)	0.002 (3)	0.030 (3)	0.015 (3)
C40	0.045 (3)	0.053 (4)	0.051 (4)	−0.008 (3)	0.024 (3)	−0.002 (3)
C41	0.049 (4)	0.055 (4)	0.092 (6)	0.009 (3)	0.032 (4)	0.007 (4)

Geometric parameters (Å, º)

Br1—C27	1.905 (4)	C18—H18	0.9500
O1—C2	1.419 (4)	C19—C20	1.386 (5)
O1—H1B	0.77 (5)	C19—H19	0.9500
O2—C31	1.358 (4)	C20—C21	1.387 (5)
O2—C32	1.434 (4)	C20—H20	0.9500

O3—C33	1.241 (5)	C21—C22	1.383 (5)
O4—C33	1.273 (5)	C21—H21	0.9500
O5—H5D	0.86 (3)	C22—H22	0.9500
O5—H5E	0.82 (3)	C23—C24	1.370 (5)
N1—C5	1.484 (5)	C23—C31	1.440 (5)
N1—C6	1.490 (5)	C24—C25	1.423 (5)
N1—C4	1.494 (4)	C24—H24	0.9500
N1—H1	1.0000	C25—C30	1.413 (5)
N2—C31	1.305 (5)	C25—C26	1.415 (5)
N2—C30	1.374 (5)	C26—C27	1.365 (5)
C1—C17	1.513 (5)	C26—H26	0.9500
C1—C23	1.526 (4)	C27—C28	1.406 (6)
C1—C2	1.572 (4)	C28—C29	1.369 (6)
C1—H1A	1.0000	C28—H28	0.9500
C2—C3	1.543 (5)	C29—C30	1.412 (5)
C2—C7	1.551 (4)	C29—H29	0.9500
C3—C4	1.525 (5)	C32—H32A	0.9800
C3—H3A	0.9900	C32—H32B	0.9800
C3—H3B	0.9900	C32—H32C	0.9800
C4—H4A	0.9900	C33—C34	1.478 (12)
C4—H4B	0.9900	C33—C34B	1.57 (3)
C5—H5A	0.9800	C34—C39	1.395 (11)
C5—H5B	0.9800	C34—C35	1.401 (10)
C5—H5C	0.9800	C35—C36	1.373 (9)
C6—H6A	0.9800	C35—H35	0.9500
C6—H6B	0.9800	C36—C37	1.405 (10)
C6—H6C	0.9800	C36—H36	0.9500
C7—C8	1.370 (6)	C37—C38	1.381 (10)
C7—C16	1.449 (5)	C37—H37	0.9500
C8—C9	1.411 (5)	C38—C39	1.385 (8)
C8—H8	0.9500	C38—H38	0.9500
C9—C10	1.359 (6)	C39—H39	0.9500
C9—H9	0.9500	C34B—C39B	1.40 (2)
C10—C11	1.408 (6)	C34B—C35B	1.41 (2)
C10—H10	0.9500	C35B—C36B	1.37 (2)
C11—C12	1.416 (6)	C35B—H35B	0.9500
C11—C16	1.440 (5)	C36B—C37B	1.41 (2)
C12—C13	1.347 (7)	C36B—H36B	0.9500
C12—H12	0.9500	C37B—C38B	1.37 (2)
C13—C14	1.408 (6)	C37B—H37B	0.9500
C13—H13	0.9500	C38B—C39B	1.384 (19)
C14—C15	1.370 (5)	C38B—H38B	0.9500
C14—H14	0.9500	C39B—H39B	0.9500
C15—C16	1.417 (5)	N3—C40	1.136 (9)
C15—H15	0.9500	C40—C41	1.448 (10)
C17—C18	1.391 (5)	C41—H41A	0.9800
C17—C22	1.393 (5)	C41—H41B	0.9800
C18—C19	1.383 (5)	C41—H41C	0.9800

C2—O1—H1B	109.5	C22—C21—C20	120.4 (3)
C31—O2—C32	116.9 (3)	C22—C21—H21	119.8
H5D—O5—H5E	104 (5)	C20—C21—H21	119.8
C5—N1—C6	110.4 (3)	C21—C22—C17	121.0 (3)
C5—N1—C4	112.7 (3)	C21—C22—H22	119.5
C6—N1—C4	109.8 (3)	C17—C22—H22	119.5
C5—N1—H1	107.9	C24—C23—C31	116.1 (3)
C6—N1—H1	107.9	C24—C23—C1	125.3 (3)
C4—N1—H1	107.9	C31—C23—C1	118.2 (3)
C31—N2—C30	117.9 (3)	C23—C24—C25	120.7 (3)
C17—C1—C23	109.9 (3)	C23—C24—H24	119.6
C17—C1—C2	110.9 (3)	C25—C24—H24	119.6
C23—C1—C2	115.5 (3)	C30—C25—C26	119.9 (3)
C17—C1—H1A	106.7	C30—C25—C24	117.8 (3)
C23—C1—H1A	106.7	C26—C25—C24	122.4 (3)
C2—C1—H1A	106.7	C27—C26—C25	118.6 (3)
O1—C2—C3	109.1 (3)	C27—C26—H26	120.7
O1—C2—C7	107.0 (3)	C25—C26—H26	120.7
C3—C2—C7	113.9 (3)	C26—C27—C28	122.5 (4)
O1—C2—C1	107.8 (3)	C26—C27—Br1	119.2 (3)
C3—C2—C1	111.2 (3)	C28—C27—Br1	118.3 (3)
C7—C2—C1	107.6 (2)	C29—C28—C27	119.1 (3)
C4—C3—C2	111.8 (3)	C29—C28—H28	120.4
C4—C3—H3A	109.2	C27—C28—H28	120.4
C2—C3—H3A	109.2	C28—C29—C30	120.6 (4)
C4—C3—H3B	109.2	C28—C29—H29	119.7
C2—C3—H3B	109.2	C30—C29—H29	119.7
H3A—C3—H3B	107.9	N2—C30—C29	118.9 (3)
N1—C4—C3	113.9 (3)	N2—C30—C25	121.9 (3)
N1—C4—H4A	108.8	C29—C30—C25	119.2 (3)
C3—C4—H4A	108.8	N2—C31—O2	119.6 (3)
N1—C4—H4B	108.8	N2—C31—C23	125.4 (3)
C3—C4—H4B	108.8	O2—C31—C23	115.0 (3)
H4A—C4—H4B	107.7	O2—C32—H32A	109.5
N1—C5—H5A	109.5	O2—C32—H32B	109.5
N1—C5—H5B	109.5	H32A—C32—H32B	109.5
H5A—C5—H5B	109.5	O2—C32—H32C	109.5
N1—C5—H5C	109.5	H32A—C32—H32C	109.5
H5A—C5—H5C	109.5	H32B—C32—H32C	109.5
H5B—C5—H5C	109.5	O3—C33—O4	124.6 (4)
N1—C6—H6A	109.5	O3—C33—C34	119.5 (5)
N1—C6—H6B	109.5	O4—C33—C34	115.9 (5)
H6A—C6—H6B	109.5	O3—C33—C34B	118.1 (11)
N1—C6—H6C	109.5	O4—C33—C34B	117.2 (11)
H6A—C6—H6C	109.5	C39—C34—C35	118.2 (9)
H6B—C6—H6C	109.5	C39—C34—C33	121.6 (9)
C8—C7—C16	117.8 (3)	C35—C34—C33	120.2 (9)

C8—C7—C2	117.3 (3)	C36—C35—C34	120.7 (8)
C16—C7—C2	124.7 (3)	C36—C35—H35	119.6
C7—C8—C9	123.5 (4)	C34—C35—H35	119.6
C7—C8—H8	118.3	C35—C36—C37	119.9 (8)
C9—C8—H8	118.3	C35—C36—H36	120.1
C10—C9—C8	119.6 (4)	C37—C36—H36	120.1
C10—C9—H9	120.2	C38—C37—C36	119.9 (7)
C8—C9—H9	120.2	C38—C37—H37	120.0
C9—C10—C11	120.4 (4)	C36—C37—H37	120.0
C9—C10—H10	119.8	C37—C38—C39	119.4 (6)
C11—C10—H10	119.8	C37—C38—H38	120.3
C10—C11—C12	120.1 (4)	C39—C38—H38	120.3
C10—C11—C16	120.5 (4)	C38—C39—C34	121.6 (7)
C12—C11—C16	119.5 (4)	C38—C39—H39	119.2
C13—C12—C11	121.7 (4)	C34—C39—H39	119.2
C13—C12—H12	119.2	C39B—C34B—C35B	119 (2)
C11—C12—H12	119.2	C39B—C34B—C33	117 (2)
C12—C13—C14	119.8 (4)	C35B—C34B—C33	124 (2)
C12—C13—H13	120.1	C36B—C35B—C34B	121 (2)
C14—C13—H13	120.1	C36B—C35B—H35B	119.6
C15—C14—C13	120.6 (4)	C34B—C35B—H35B	119.6
C15—C14—H14	119.7	C35B—C36B—C37B	118 (2)
C13—C14—H14	119.7	C35B—C36B—H36B	121.2
C14—C15—C16	121.8 (4)	C37B—C36B—H36B	121.2
C14—C15—H15	119.1	C38B—C37B—C36B	120 (2)
C16—C15—H15	119.1	C38B—C37B—H37B	119.8
C15—C16—C11	116.7 (3)	C36B—C37B—H37B	119.8
C15—C16—C7	125.1 (3)	C37B—C38B—C39B	120.0 (17)
C11—C16—C7	118.2 (3)	C37B—C38B—H38B	120.0
C18—C17—C22	118.1 (3)	C39B—C38B—H38B	120.0
C18—C17—C1	120.3 (3)	C38B—C39B—C34B	119.7 (19)
C22—C17—C1	121.5 (3)	C38B—C39B—H39B	120.2
C19—C18—C17	121.0 (3)	C34B—C39B—H39B	120.2
C19—C18—H18	119.5	N3—C40—C41	177.7 (8)
C17—C18—H18	119.5	C40—C41—H41A	109.5
C18—C19—C20	120.4 (3)	C40—C41—H41B	109.5
C18—C19—H19	119.8	H41A—C41—H41B	109.5
C20—C19—H19	119.8	C40—C41—H41C	109.5
C19—C20—C21	119.1 (4)	H41A—C41—H41C	109.5
C19—C20—H20	120.4	H41B—C41—H41C	109.5
C21—C20—H20	120.4		

C17—C1—C2—O1	54.5 (3)	C17—C1—C23—C31	87.0 (4)
C23—C1—C2—O1	−71.4 (3)	C2—C1—C23—C31	−146.7 (3)
C17—C1—C2—C3	174.1 (3)	C31—C23—C24—C25	2.2 (5)
C23—C1—C2—C3	48.2 (4)	C1—C23—C24—C25	175.4 (3)
C17—C1—C2—C7	−60.6 (3)	C23—C24—C25—C30	1.3 (5)
C23—C1—C2—C7	173.6 (3)	C23—C24—C25—C26	−178.0 (3)

O1—C2—C3—C4	−76.1 (3)	C30—C25—C26—C27	0.6 (5)
C7—C2—C3—C4	43.4 (4)	C24—C25—C26—C27	179.9 (3)
C1—C2—C3—C4	165.1 (3)	C25—C26—C27—C28	3.2 (6)
C5—N1—C4—C3	65.3 (4)	C25—C26—C27—Br1	−177.0 (3)
C6—N1—C4—C3	−171.3 (3)	C26—C27—C28—C29	−4.1 (6)
C2—C3—C4—N1	165.5 (3)	Br1—C27—C28—C29	176.1 (3)
O1—C2—C7—C8	−8.1 (4)	C27—C28—C29—C30	1.1 (6)
C3—C2—C7—C8	−128.8 (3)	C31—N2—C30—C29	−179.3 (4)
C1—C2—C7—C8	107.5 (3)	C31—N2—C30—C25	1.4 (5)
O1—C2—C7—C16	176.3 (3)	C28—C29—C30—N2	−176.9 (4)
C3—C2—C7—C16	55.6 (4)	C28—C29—C30—C25	2.4 (6)
C1—C2—C7—C16	−68.1 (4)	C26—C25—C30—N2	176.0 (3)
C16—C7—C8—C9	−0.1 (6)	C24—C25—C30—N2	−3.3 (5)
C2—C7—C8—C9	−175.9 (3)	C26—C25—C30—C29	−3.3 (6)
C7—C8—C9—C10	−0.9 (6)	C24—C25—C30—C29	177.4 (3)
C8—C9—C10—C11	1.2 (6)	C30—N2—C31—O2	−177.1 (3)
C9—C10—C11—C12	178.9 (4)	C30—N2—C31—C23	2.6 (5)
C9—C10—C11—C16	−0.5 (6)	C32—O2—C31—N2	2.4 (5)
C10—C11—C12—C13	−179.1 (5)	C32—O2—C31—C23	−177.3 (3)
C16—C11—C12—C13	0.4 (7)	C24—C23—C31—N2	−4.4 (5)
C11—C12—C13—C14	−0.3 (8)	C1—C23—C31—N2	−178.2 (3)
C12—C13—C14—C15	0.1 (8)	C24—C23—C31—O2	175.3 (3)
C13—C14—C15—C16	0.0 (7)	C1—C23—C31—O2	1.5 (5)
C14—C15—C16—C11	0.1 (6)	O3—C33—C34—C39	21 (2)
C14—C15—C16—C7	179.7 (4)	O4—C33—C34—C39	−157.5 (13)
C10—C11—C16—C15	179.2 (4)	O3—C33—C34—C35	−159.2 (14)
C12—C11—C16—C15	−0.3 (6)	O4—C33—C34—C35	22 (2)
C10—C11—C16—C7	−0.5 (5)	C39—C34—C35—C36	−3 (3)
C12—C11—C16—C7	−179.9 (4)	C33—C34—C35—C36	177.0 (13)
C8—C7—C16—C15	−178.9 (4)	C34—C35—C36—C37	6 (2)
C2—C7—C16—C15	−3.3 (5)	C35—C36—C37—C38	−6.6 (17)
C8—C7—C16—C11	0.7 (5)	C36—C37—C38—C39	3.6 (14)
C2—C7—C16—C11	176.3 (3)	C37—C38—C39—C34	−0.6 (16)
C23—C1—C17—C18	−135.1 (3)	C35—C34—C39—C38	0 (2)
C2—C1—C17—C18	95.9 (4)	C33—C34—C39—C38	180.0 (12)
C23—C1—C17—C22	46.4 (4)	O3—C33—C34B—C39B	4 (6)
C2—C1—C17—C22	−82.5 (4)	O4—C33—C34B—C39B	−179 (3)
C22—C17—C18—C19	0.1 (5)	O3—C33—C34B—C35B	−175 (5)
C1—C17—C18—C19	−178.4 (3)	O4—C33—C34B—C35B	2 (7)
C17—C18—C19—C20	0.4 (6)	C39B—C34B—C35B—C36B	4 (8)
C18—C19—C20—C21	−0.7 (6)	C33—C34B—C35B—C36B	−177 (4)
C19—C20—C21—C22	0.4 (6)	C34B—C35B—C36B—C37B	−14 (6)
C20—C21—C22—C17	0.1 (6)	C35B—C36B—C37B—C38B	19 (5)
C18—C17—C22—C21	−0.3 (6)	C36B—C37B—C38B—C39B	−15 (4)
C1—C17—C22—C21	178.2 (3)	C37B—C38B—C39B—C34B	5 (5)
C17—C1—C23—C24	−86.1 (4)	C35B—C34B—C39B—C38B	1 (8)
C2—C1—C23—C24	40.2 (5)	C33—C34B—C39B—C38B	−178 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O5	0.77	1.94	2.691 (4)	163
O5—H5*D*···O4^{i}	0.86 (3)	1.89 (3)	2.738 (4)	168 (6)
O5—H5*E*···O3	0.82 (3)	1.92 (3)	2.734 (4)	172 (6)
N1—H1···O4^{i}	1.00	1.62	2.619 (4)	178
C5—H5*C*···Br1^{ii}	0.98	3.13	3.964 (4)	144
C6—H6*A*···O3	0.98	2.63	3.562 (6)	159
C28—H28···O5^{ii}	0.95	2.66	3.498 (5)	148

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