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The methanol and ethanol solvates of 4-glutarato-*N*,*N*-diisopropyltryptamine

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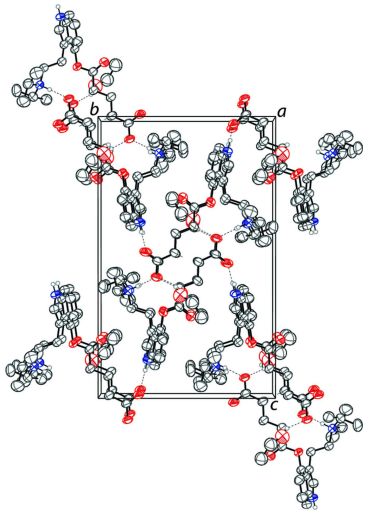
The solid-state structures of two solvated forms of 4-glutarato-*N*,*N*-diisopropyltryptamine were determined by single-crystal X-ray diffraction, namely, 5-[(3-{2-[bis(propan-2-yl)azaniumyl]ethyl}-1*H*-indol-4-yl)oxy]-5-oxopentanoate methanol monosolvate, $C_{21}H_{30}N_2O_4\cdot CH_3OH$, and the analogous ethanol monosolvate, $C_{21}H_{30}N_2O_4\cdot C_2H_6O$. In both compounds, the 4-glutarato-*N*,*N*-diisopropyltryptamine exists as a zwitterion with a protonated tertiary ammonium and a deprotonated glutarato carboxylate. The tryptamine zwitterions and alcohol solvates in both structures combine to produce near identical hydrogen-bonding networks, with N—H···O and O—H···O hydrogen bonds joining the molecules together in two-dimensional networks parallel to the (100) plane.

1. Chemical context

Psychedelic compounds continue to be a major research focus for treating conditions including depression, post-traumatic stress disorder (PTSD), Alzheimer's disease, and chronic pain (Carhart-Harris & Goodwin, 2017; Krediet *et al.*, 2020; Vann Jones & O'Kelly, 2020; Ramaekers *et al.*, 2021). Tryptamine compounds with chemical structures resembling that of the active product of magic mushrooms, psilocin (4-hydroxy-*N*,*N*-dimethyltryptamine; 4-HO-DMT), are of particular interest. This is due not just to their structural similarities to the neurotransmitter serotonin (5-hydroxytryptamine; 5-HT), but because many have desirable drug characteristics including oral availability, lowered susceptibility to monoamine oxidase (MAO) degradation, and short duration of action (Kuypers *et al.*, 2019). The synthesis of prodrugs that undergo hydrolysis to produce 4-hydroxy derivatives of dialkyltryptamines are of increasing interest (Klein *et al.*, 2021; Chadeayne *et al.*, 2019*a*; Chadeayne, Pham, Reid *et al.*, 2020; Naeem *et al.*, 2022).

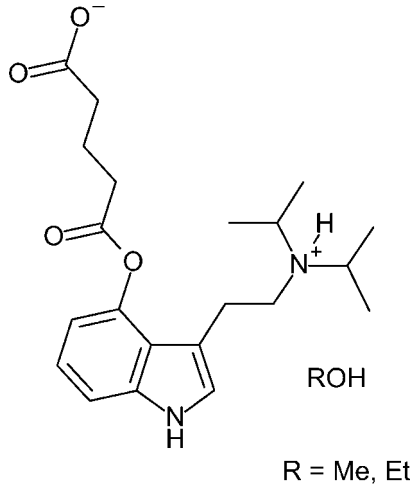
4-Hydroxy-*N*,*N*-diisopropyltryptamine (4-HO-DiPT) is one example of a psilocin analog, first synthesized in 1977, in which both methyl groups on the ethylamino moiety of psilocin are replaced with isopropyl groups (Repke *et al.*, 1977). In early 2022, 4-HO-DiPT along with four other psychedelics were part of a proposal issued by the US Drug Enforcement Administration (DEA), requesting comments on reclassifying these compounds to Schedule I of the Controlled Substance Act. Due to a strong public response, the DEA withdrew the proposal before the hearing, which was scheduled for August (US DEA, January 14 & July 6, 2022*a*,*b*).

4-HO-DiPT is a serotonin-2A (5-HT_{2A}) receptor agonist that, like psilocin, produces a head-twitch response (HTR) in



mice, indicating its competence in producing psychedelic effects (Halberstadt *et al.*, 2020). 4-HO-DiPT also interacts with the serotonin transporter (SERT) with IC_{50} values in the low micromolar range, similar to 3,4-methylenedioxymethamphetamine (MDMA) (Rickli *et al.*, 2016). 4-HO-DiPT has been reported as orally active at a 15–20 mg dose, with its profound psychedelic effects beginning within 15 minutes and lasting about 2–3 h (Shulgin & Shulgin, 2017).

4-HO-DiPT glutarate, a 'hemiester' prodrug of 4-HO-DiPT has been reported in the patent literature (Bryson, 2022). We have previously published work characterizing tryptamine compounds, highlighting the importance of single-crystal X-ray diffraction studies when characterizing tryptamine salts because they can occur in a variety of forms that are often not appreciated by other means of characterization (Chadeayne *et al.*, 2019*a*,*b*; Chadeayne, Pham, Golen *et al.*, 2020; Sammeta *et al.*, 2020; Pham *et al.*, 2021; Naeem *et al.*, 2022). To this end, we synthesized 4-glutarato-*N*,*N*-diisopropyltrypamine and report herein two crystalline forms of the compound as both its methanol and ethanol solvates.



2. Structural commentary

In the solid state, the compound exists as a zwitterion, with a protonated tertiary ammonium group and a deprotonated carboxylate of the glutarato group. Both of the solvate structures possess one zwitterionic molecule and one alcohol molecule in the asymmetric unit (Fig. 1). In the ethanol solvate, the alcohol molecule is disordered over two orientations in a 0.531 (11):0.469 (11) ratio. Both solvates have near planar indole units with r.m.s. deviations from planarity of 0.009 and 0.016 Å for the methanol and ethanol solvates, respectively. The glutarato units are also close to planar with r.m.s. deviations of only 0.061 and 0.071 Å. In both cases, the glutarato unit is nearly orthogonal to the indole plane, showing plane-to-plane twists of 90.99 (6) and 94.21 (8)°. Likewise, the ethylamino arms are nearly orthogonal to the indole plane with C7—C8—C9—C10 angles of 90.2 (2) and 86.1 (3)°. Both ethylamino arms demonstrate *anti* configurations, with C8—C9—C10—N2 angles of 179.92 (14) and 180.0 (2)°. In both structures, the glutarato and ethylamino

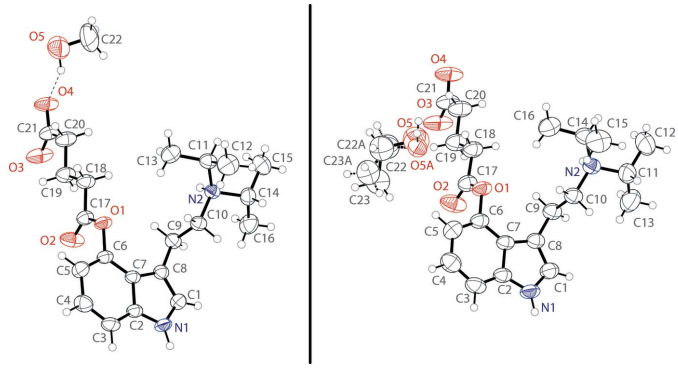


Figure 1
The molecular structures of 4-glutarato-*N*,*N*-diisopropyltryptamine as both its methanol (left) and ethanol (right) solvate, showing atomic labeling. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines. Dashed bonds indicate the minor occupancy disordered component in the ethanol solvate.

arms are turned to opposite sides of the indole. This differs from the structures observed in other zwitterionic indoles where intramolecular hydrogen bonding leads to two groups being on the same side of the aromatic rings (Naeem *et al.*, 2022). The nature of the groups in this compound only allows for intermolecular interactions (*vide infra*) and having the groups on opposite sides of the indole is sterically preferred.

3. Supramolecular features

In both crystals, the zwitterionic molecules and alcohol solvents are held together by N^+—H···O^- and O—H···O hydrogen bonds that produce infinite two-dimensional networks parallel to the (100) plane. The most significant hydrogen bonds are N2—H2···O4 bonds between the diisopropyltryptammonium cation and the carboxylate anion of another zwitterionic molecule. These interactions form centrosymmetrical dimers, which form rings with graph-set notation of $R_2^2(28)$ (Etter *et al.*, 1990). These dimers are shown in Fig. 2. The dimers are joined together through N1—

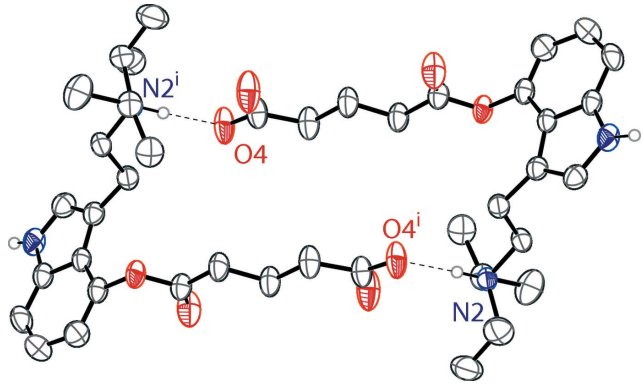


Figure 2
The ring formed by the dimerization of two zwitterionic 4-glutarato-*N*,*N*-diisopropyltryptamine molecules with graph set notation of $R_2^2(28)$. The image shown is from the methanol solvate. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding are omitted for clarity. Symmetry code: (i) $1-x, 1-y, 1-z$.

Table 1
Hydrogen-bond geometry (Å, °) for the methanol solvate.

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O4	1.00 (1)	1.83 (2)	2.748 (2)	151 (3)
N2—H2···O4^{i}	0.90 (1)	1.81 (1)	2.7154 (19)	177 (2)
N1—H1···O3^{ii}	0.86 (1)	1.99 (1)	2.773 (2)	151 (2)

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

H1···O3 hydrogen bonds between the indole nitrogen and the other carboxylate oxygen. The alcohol oxygens also hydrogen bond to the carboxylate anion through O5—H5···O4 bonds (Tables 1 and 2). The two structures demonstrate near identical hydrogen-bonding networks in the solid state, which can be seen in their packing diagrams (Fig. 3).

4. Database survey

There are three reported tryptamine structures possessing isopropyl groups on the ethylamino arm, all of which are *N*-methyl-*N*-isopropyl derivatives: *N*-methyl-*N*-isopropyltryptammonium hydrofumarate (Chadeayne, Pham, Golen *et al.*, 2019: RONSOF) as well as the hydrofumarate (Chadeayne, Pham, Golen *et al.*, 2019: RONSUL) and fumarate (Chadeayne, Pham, Golen *et al.*, 2020: TUFQAP) of 4-hydroxy-*N*-methyl-*N*-isopropyltryptamine. There are six structures of 4-substituted esters of tryptamines in the literature, all of which are 4-acetoxy derivatives: the hydrofumarate (Chadeayne *et al.*, 2019*a*: HOCJUH) and fumarate (Chadeayne *et al.*, 2019*b*: XOFDOO) of psilacetin (4-acetoxy-*N*,*N*-dimethyltryptamine), 4-acetoxy-*N*-methyl-*N*-ethyltryptammonium hydrofumarate (Pham *et al.*, 2021: OJIQIK), 4-acetoxy-*N*-methyl-*N*-allyltryptammonium hydrofumarate (Pham *et al.*, 2021: OJIQOQ), 4-acetoxy-*N*,*N*-diallyltryptammonium fumarate fumaric acid (Pham *et al.*, 2021: OJIQUW), and 4-acetoxy-*N*,*N*,*N*-trimethyltryptammonium iodide (Chadeayne, Pham, Reid *et al.*, 2020: XUXDUS). There are two tryptamine zwitterions reported in the literature, those being the natural products baeocystin, 4-phosphoryloxy-*N*-methyltryptamine (Naeem *et al.*, 2022), and psilocybin, 4-phosphoryloxy-*N*,*N*-dimethyltryptamine (Weber & Petcher, 1974; PSILOC; Sherwood *et al.*, 2022: TAVZID, TAVZID01; Greenan *et al.*, 2020; OKOKAD).

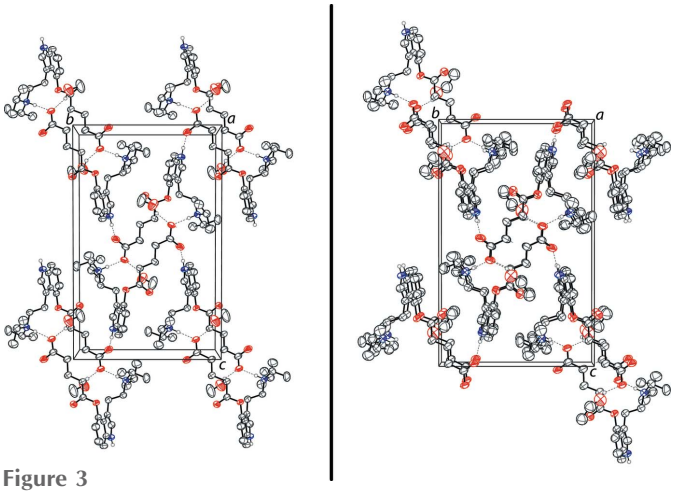


Figure 3
The crystal packing of the methanol solvate (left) and the ethanol solvate (right) of 4-glutarato-*N*,*N*-diisopropyltryptamine, both shown along the *a*-axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding are omitted for clarity.

Table 2
Hydrogen-bond geometry (Å, °) for the ethanol solvate.

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O4^{i}	0.90 (3)	1.79 (3)	2.686 (3)	177 (3)
N1—H1···O3^{ii}	0.85 (1)	1.91 (1)	2.751 (3)	167 (3)
O5—H5A···O4^{iii}	0.82	1.97	2.692 (10)	147
O5A—H5AA···O4^{iii}	0.82	1.95	2.732 (6)	160

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

5. Synthesis and crystallization

112 mg of 4-hydroxy-*N*,*N*-diisopropyltryptamine (1 mmol) were dissolved in 5 mL of chloroform. 0.3 mL of triethylamine (5 mmol) followed by 490 mg of glutaric anhydride (10 mmol) were then added to the solution. The mixture was stirred at room temperature for 30 minutes, resulting in a precipitate which was isolated *via* filtration. The precipitate was triturated with tetrahydrofuran and washed with chloroform to obtain 73 mg of white powder (65% yield).

^{1}H NMR (400 MHz, DMSO-d_6): δ 11.02 (*s*, 1H, N*H*), 7.22 (*d*, *J* = 8.1 Hz, 1H, Ar*H*), 7.16 (*d*, *J* = 2.3 Hz, 1H, Ar*H*), 7.02 (*t*, *J* = 7.9 Hz, 1H, Ar*H*), 6.64 (*d*, *J* = 7.5 Hz, 1H, Ar*H*), 3.10 (*sept*, *J* = 6.5 Hz, 2H, C*H*), 2.77–2.63 (*m*, 6H, CH_2), 2.31 (*t*, *J* = 7.2 Hz, 2H, CH_2), 1.88 (*t*, *J* = 7.2 Hz, 2H, CH_2), 1.00 (*d*, *J* = 6.6 Hz, 12H, CH_3).

The powder was recrystallized from boiling methanol to yield single crystals of the methanol solvate suitable for X-ray diffraction analysis. Slow evaporation of an ethanol solution of the powder produced single crystals of the ethanol solvate suitable for X-ray diffraction analysis.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. In the methanol solvate, hydrogen atoms H1, H2 and H5*A* were found in a difference-Fourier map and in the ethanol solvate, hydrogen atoms H1 and H2 were found in a difference-Fourier map. These hydrogens were refined isotropically, using DFIX restraints with N—H(indole) distances of 0.87 (1) Å, N—H(ammonium) distances of 0.90 (1) Å, and O—H distances of 0.99 (1) Å. Isotropic displacement parameters were set to 1.2U_{eq} of the parent nitrogen atoms and 1.5U_{eq} of the parent oxygen atom. All other hydrogens were placed in calculated positions [C—H = 0.93 Å (sp^2), 0.97 Å (CH_2), 0.96 Å (CH_3)]. The hydrogen atoms in the disordered ethanol molecule were placed in calculated positions [O—H = 0.82 Å]. Isotropic displacement parameters were set to 1.2U_{eq} of the parent carbon atoms and 1.5U_{eq} of the parent oxygen atoms.

Table 3
Experimental details.

	Methanol solvate	Ethanol solvate
Crystal data		
Chemical formula	$C_{21}H_{30}N_2O_4{\cdot}CH_4O$	$C_{21}H_{30}N_2O_4{\cdot}C_2H_6O$
M_r	406.51	420.54
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
Temperature (K)	297	297
a, b, c (Å)	7.9531 (5), 13.4224 (7), 21.2015 (11)	8.0087 (12), 13.7968 (17), 21.878 (3)
β (°)	92.484 (2)	90.749 (4)
V (Å^3)	2261.1 (2)	2417.2 (5)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.08	0.08
Crystal size (mm)	0.22 × 0.21 × 0.20	0.30 × 0.27 × 0.22
Data collection		
Diffractometer	Bruker D8 Venture CMOS	Bruker D8 Venture CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2021)	Multi-scan (*SADABS*; Bruker, 2021)
T_{min}, T_{max}	0.718, 0.745	0.692, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	61210, 4304, 3531	37412, 4461, 3038
R_{int}	0.039	0.055
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.610	0.604
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.143, 1.03	0.060, 0.176, 1.04
No. of reflections	4304	4461
No. of parameters	279	313
No. of restraints	3	46
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
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.46, −0.39	0.35, −0.46

Computer programs: *APEX4* and *SAINT* (Bruker, 2021), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009), and *publCIF* (Westrip, 2010).

Acknowledgements

Financial statements and conflict of interest: This study was funded by CaaMTech, Inc. ARC reports an ownership interest in CaaMTech, Inc., which owns US and worldwide patent applications, covering new tryptamine compounds, compositions, formulations, novel crystalline forms, and methods of making and using the same.

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supporting information

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The methanol and ethanol solvates of 4-glutarato-*N*,*N*-diisopropyltryptamine

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Computing details

For both structures, data collection: *APEX4* (Bruker, 2021); cell refinement: *SAINT* (Bruker, 2021); data reduction: *SAINT* (Bruker, 2021); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

5-[(3-{2-[Bis(propan-2-yl)azaniumyl]ethyl}-1*H*-indol-4-yl)oxy]-5-oxopentanoate methanol monosolvate (I)

Crystal data

$C_{21}H_{30}N_2O_4{\cdot}CH_4O$
M_r = 406.51
Monoclinic, $P2_1/c$
a = 7.9531 (5) Å
b = 13.4224 (7) Å
c = 21.2015 (11) Å
β = 92.484 (2)°
V = 2261.1 (2) Å^3
Z = 4
$F(000)$ = 880
D_x = 1.194 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9859 reflections
θ = 3.0–25.6°
μ = 0.08 mm^{-1}
T = 297 K
Block, colourless
0.22 × 0.21 × 0.20 mm

Data collection

Bruker D8 Venture CMOS diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2021)
T_{min} = 0.718, T_{max} = 0.745
61210 measured reflections
4304 independent reflections
3531 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 25.7°, θ_{min} = 3.0°
h = −9→9
k = −16→16
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.143
S = 1.03
4304 reflections
279 parameters
3 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2 + 1.0658P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0049 (16)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.17552 (17)	0.35382 (9)	0.29846 (5)	0.0421 (3)
O2	0.2385 (2)	0.51443 (11)	0.28621 (6)	0.0675 (5)
O3	0.3521 (2)	0.72404 (12)	0.48361 (7)	0.0730 (5)
O4	0.2931 (2)	0.66404 (11)	0.57648 (6)	0.0621 (4)
N1	0.4481 (2)	0.26115 (12)	0.11776 (7)	0.0454 (4)
N2	0.61740 (18)	0.15461 (10)	0.37628 (6)	0.0362 (3)
C1	0.5573 (2)	0.25778 (14)	0.16893 (9)	0.0446 (4)
H1A	0.669647	0.238884	0.167694	0.053*
C2	0.2940 (2)	0.29148 (13)	0.13645 (8)	0.0386 (4)
C3	0.1426 (3)	0.30411 (15)	0.10225 (8)	0.0481 (5)
H3	0.134321	0.291932	0.059041	0.058*
C4	0.0058 (3)	0.33510 (17)	0.13421 (9)	0.0539 (5)
H4	−0.096839	0.343720	0.112189	0.065*
C5	0.0169 (2)	0.35412 (15)	0.19936 (9)	0.0489 (5)
H5	−0.077222	0.375904	0.219944	0.059*
C6	0.1662 (2)	0.34057 (12)	0.23242 (8)	0.0375 (4)
C7	0.3099 (2)	0.30850 (12)	0.20252 (7)	0.0348 (4)
C8	0.4801 (2)	0.28580 (12)	0.22217 (8)	0.0381 (4)
C9	0.5583 (2)	0.27857 (13)	0.28772 (8)	0.0410 (4)
H9A	0.501378	0.323224	0.315755	0.049*
H9B	0.675960	0.297597	0.287599	0.049*
C10	0.5426 (2)	0.17173 (13)	0.31047 (8)	0.0387 (4)
H10A	0.424472	0.153645	0.309562	0.046*
H10B	0.598183	0.128085	0.281434	0.046*
C11	0.4906 (2)	0.10852 (14)	0.41929 (9)	0.0452 (4)
H11	0.547211	0.100130	0.460941	0.054*
C13	0.3444 (3)	0.17846 (17)	0.42725 (11)	0.0581 (5)
H13A	0.385521	0.241480	0.442784	0.087*
H13B	0.269327	0.150484	0.456783	0.087*
H13C	0.285274	0.187901	0.387253	0.087*
C12	0.4347 (4)	0.00616 (17)	0.39679 (13)	0.0723 (7)
H12A	0.530499	−0.037275	0.396077	0.108*
H12B	0.383512	0.011193	0.355068	0.108*
H12C	0.354637	−0.020317	0.424972	0.108*
C14	0.7823 (2)	0.09675 (15)	0.37460 (9)	0.0471 (5)
H14	0.759363	0.032539	0.353974	0.057*
C15	0.8546 (3)	0.0769 (2)	0.44071 (11)	0.0712 (7)
H15A	0.783385	0.030922	0.461678	0.107*
H15B	0.861037	0.138280	0.463944	0.107*

H15C	0.965311	0.048954	0.438396	0.107*
C16	0.9078 (3)	0.15321 (19)	0.33620 (12)	0.0632 (6)
H16A	0.862701	0.161671	0.293803	0.095*
H16B	1.011172	0.116333	0.335554	0.095*
H16C	0.929013	0.217350	0.354941	0.095*
C17	0.2114 (2)	0.44694 (13)	0.32012 (8)	0.0409 (4)
C18	0.2177 (3)	0.44943 (14)	0.39084 (8)	0.0483 (5)
H18A	0.306025	0.404959	0.406611	0.058*
H18B	0.111785	0.424768	0.405632	0.058*
C19	0.2497 (3)	0.55192 (15)	0.41772 (9)	0.0522 (5)
H19A	0.162315	0.596552	0.401363	0.063*
H19B	0.356367	0.576115	0.403349	0.063*
C20	0.2538 (3)	0.55546 (15)	0.48899 (9)	0.0536 (5)
H20A	0.332027	0.505235	0.505171	0.064*
H20B	0.143059	0.538020	0.502980	0.064*
C21	0.3041 (3)	0.65529 (15)	0.51767 (9)	0.0474 (5)
O5	0.1577 (3)	0.53742 (16)	0.66260 (11)	0.0928 (6)
H5A	0.224 (4)	0.563 (2)	0.6271 (11)	0.111*
C22	0.2529 (6)	0.4611 (3)	0.6828 (2)	0.1272 (15)
H22A	0.184260	0.413329	0.703499	0.191*
H22B	0.339437	0.484832	0.712036	0.191*
H22C	0.303585	0.430125	0.647521	0.191*
H2	0.644 (2)	0.2153 (9)	0.3923 (9)	0.045 (5)*
H1	0.475 (3)	0.2488 (16)	0.0796 (6)	0.057 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0613 (8)	0.0378 (6)	0.0278 (6)	−0.0046 (6)	0.0104 (5)	−0.0059 (5)
O2	0.1172 (14)	0.0483 (8)	0.0375 (7)	−0.0279 (9)	0.0073 (8)	0.0011 (6)
O3	0.1127 (14)	0.0647 (10)	0.0436 (8)	−0.0400 (9)	0.0275 (8)	−0.0135 (7)
O4	0.1033 (12)	0.0526 (8)	0.0311 (7)	−0.0267 (8)	0.0110 (7)	−0.0100 (6)
N1	0.0607 (10)	0.0477 (9)	0.0289 (7)	0.0035 (7)	0.0133 (7)	−0.0045 (6)
N2	0.0427 (8)	0.0336 (7)	0.0321 (7)	−0.0030 (6)	−0.0009 (6)	−0.0024 (6)
C1	0.0484 (10)	0.0436 (10)	0.0421 (10)	0.0040 (8)	0.0068 (8)	−0.0017 (8)
C2	0.0537 (10)	0.0338 (8)	0.0286 (8)	−0.0027 (7)	0.0058 (7)	−0.0025 (6)
C3	0.0624 (12)	0.0538 (11)	0.0278 (8)	−0.0041 (9)	−0.0028 (8)	−0.0026 (8)
C4	0.0502 (11)	0.0655 (13)	0.0452 (11)	−0.0023 (10)	−0.0074 (9)	0.0017 (9)
C5	0.0455 (10)	0.0558 (11)	0.0460 (11)	0.0010 (9)	0.0075 (8)	−0.0030 (9)
C6	0.0500 (10)	0.0347 (8)	0.0283 (8)	−0.0044 (7)	0.0073 (7)	−0.0033 (6)
C7	0.0476 (9)	0.0301 (8)	0.0266 (8)	−0.0030 (7)	0.0026 (7)	−0.0017 (6)
C8	0.0480 (10)	0.0333 (8)	0.0331 (8)	−0.0006 (7)	0.0028 (7)	−0.0002 (7)
C9	0.0477 (10)	0.0379 (9)	0.0370 (9)	−0.0015 (7)	−0.0027 (7)	−0.0032 (7)
C10	0.0440 (9)	0.0398 (9)	0.0318 (8)	−0.0042 (7)	−0.0027 (7)	−0.0033 (7)
C11	0.0531 (11)	0.0423 (10)	0.0404 (9)	−0.0060 (8)	0.0054 (8)	0.0046 (8)
C13	0.0523 (12)	0.0638 (13)	0.0592 (13)	−0.0052 (10)	0.0121 (10)	−0.0035 (10)
C12	0.0905 (18)	0.0434 (12)	0.0845 (17)	−0.0185 (12)	0.0207 (14)	0.0013 (11)
C14	0.0463 (10)	0.0472 (10)	0.0475 (10)	0.0064 (8)	−0.0030 (8)	−0.0041 (8)

C15	0.0655 (15)	0.0859 (17)	0.0609 (14)	0.0185 (13)	−0.0131 (11)	0.0064 (12)
C16	0.0448 (11)	0.0772 (16)	0.0678 (14)	0.0022 (10)	0.0058 (10)	−0.0027 (12)
C17	0.0503 (10)	0.0382 (9)	0.0349 (9)	−0.0052 (8)	0.0075 (7)	−0.0045 (7)
C18	0.0719 (13)	0.0414 (10)	0.0319 (9)	−0.0056 (9)	0.0054 (8)	−0.0059 (7)
C19	0.0747 (14)	0.0454 (11)	0.0367 (10)	−0.0082 (9)	0.0036 (9)	−0.0102 (8)
C20	0.0731 (14)	0.0505 (11)	0.0380 (10)	−0.0169 (10)	0.0104 (9)	−0.0105 (8)
C21	0.0573 (11)	0.0509 (11)	0.0349 (9)	−0.0159 (9)	0.0107 (8)	−0.0100 (8)
O5	0.0849 (13)	0.0864 (14)	0.1090 (16)	0.0042 (11)	0.0245 (11)	0.0304 (12)
C22	0.152 (4)	0.073 (2)	0.159 (4)	0.045 (2)	0.036 (3)	0.038 (2)

Geometric parameters (Å, º)

O1—C6	1.4100 (19)	C11—C12	1.515 (3)
O1—C17	1.358 (2)	C13—H13A	0.9600
O2—C17	1.182 (2)	C13—H13B	0.9600
O3—C21	1.242 (2)	C13—H13C	0.9600
O4—C21	1.259 (2)	C12—H12A	0.9600
N1—C1	1.361 (2)	C12—H12B	0.9600
N1—C2	1.366 (2)	C12—H12C	0.9600
N1—H1	0.862 (9)	C14—H14	0.9800
N2—C10	1.510 (2)	C14—C15	1.515 (3)
N2—C11	1.520 (2)	C14—C16	1.518 (3)
N2—C14	1.526 (2)	C15—H15A	0.9600
N2—H2	0.903 (9)	C15—H15B	0.9600
C1—H1A	0.9300	C15—H15C	0.9600
C1—C8	1.361 (2)	C16—H16A	0.9600
C2—C3	1.389 (3)	C16—H16B	0.9600
C2—C7	1.420 (2)	C16—H16C	0.9600
C3—H3	0.9300	C17—C18	1.498 (2)
C3—C4	1.371 (3)	C18—H18A	0.9700
C4—H4	0.9300	C18—H18B	0.9700
C4—C5	1.404 (3)	C18—C19	1.506 (3)
C5—H5	0.9300	C19—H19A	0.9700
C5—C6	1.364 (3)	C19—H19B	0.9700
C6—C7	1.399 (2)	C19—C20	1.511 (3)
C7—C8	1.431 (3)	C20—H20A	0.9700
C8—C9	1.501 (2)	C20—H20B	0.9700
C9—H9A	0.9700	C20—C21	1.518 (3)
C9—H9B	0.9700	O5—H5A	0.998 (10)
C9—C10	1.520 (2)	O5—C22	1.334 (4)
C10—H10A	0.9700	C22—H22A	0.9600
C10—H10B	0.9700	C22—H22B	0.9600
C11—H11	0.9800	C22—H22C	0.9600
C11—C13	1.510 (3)		

C17—O1—C6	116.95 (13)	H13B—C13—H13C	109.5
C1—N1—C2	109.15 (14)	C11—C12—H12A	109.5
C1—N1—H1	124.4 (15)	C11—C12—H12B	109.5

C2—N1—H1	126.4 (15)	C11—C12—H12C	109.5
C10—N2—C11	111.80 (14)	H12A—C12—H12B	109.5
C10—N2—C14	111.11 (13)	H12A—C12—H12C	109.5
C10—N2—H2	106.6 (13)	H12B—C12—H12C	109.5
C11—N2—C14	113.58 (14)	N2—C14—H14	108.3
C11—N2—H2	106.8 (13)	C15—C14—N2	111.09 (16)
C14—N2—H2	106.4 (13)	C15—C14—H14	108.3
N1—C1—H1A	124.6	C15—C14—C16	110.61 (19)
N1—C1—C8	110.89 (17)	C16—C14—N2	110.14 (16)
C8—C1—H1A	124.6	C16—C14—H14	108.3
N1—C2—C3	130.79 (16)	C14—C15—H15A	109.5
N1—C2—C7	106.95 (15)	C14—C15—H15B	109.5
C3—C2—C7	122.25 (17)	C14—C15—H15C	109.5
C2—C3—H3	121.1	H15A—C15—H15B	109.5
C4—C3—C2	117.88 (17)	H15A—C15—H15C	109.5
C4—C3—H3	121.1	H15B—C15—H15C	109.5
C3—C4—H4	119.2	C14—C16—H16A	109.5
C3—C4—C5	121.60 (18)	C14—C16—H16B	109.5
C5—C4—H4	119.2	C14—C16—H16C	109.5
C4—C5—H5	120.1	H16A—C16—H16B	109.5
C6—C5—C4	119.84 (18)	H16A—C16—H16C	109.5
C6—C5—H5	120.1	H16B—C16—H16C	109.5
C5—C6—O1	120.02 (16)	O1—C17—C18	110.89 (14)
C5—C6—C7	121.21 (16)	O2—C17—O1	122.74 (16)
C7—C6—O1	118.69 (15)	O2—C17—C18	126.32 (17)
C2—C7—C8	107.19 (15)	C17—C18—H18A	108.9
C6—C7—C2	117.21 (16)	C17—C18—H18B	108.9
C6—C7—C8	135.60 (15)	C17—C18—C19	113.38 (16)
C1—C8—C7	105.81 (15)	H18A—C18—H18B	107.7
C1—C8—C9	124.48 (17)	C19—C18—H18A	108.9
C7—C8—C9	129.19 (15)	C19—C18—H18B	108.9
C8—C9—H9A	110.0	C18—C19—H19A	108.8
C8—C9—H9B	110.0	C18—C19—H19B	108.8
C8—C9—C10	108.57 (14)	C18—C19—C20	113.78 (16)
H9A—C9—H9B	108.4	H19A—C19—H19B	107.7
C10—C9—H9A	110.0	C20—C19—H19A	108.8
C10—C9—H9B	110.0	C20—C19—H19B	108.8
N2—C10—C9	113.72 (13)	C19—C20—H20A	108.5
N2—C10—H10A	108.8	C19—C20—H20B	108.5
N2—C10—H10B	108.8	C19—C20—C21	114.98 (16)
C9—C10—H10A	108.8	H20A—C20—H20B	107.5
C9—C10—H10B	108.8	C21—C20—H20A	108.5
H10A—C10—H10B	107.7	C21—C20—H20B	108.5
N2—C11—H11	107.3	O3—C21—O4	122.88 (17)
C13—C11—N2	110.38 (15)	O3—C21—C20	120.35 (16)
C13—C11—H11	107.3	O4—C21—C20	116.77 (17)
C13—C11—C12	112.46 (19)	C22—O5—H5A	101 (2)
C12—C11—N2	111.93 (16)	O5—C22—H22A	109.5

C12—C11—H11	107.3	O5—C22—H22B	109.5
C11—C13—H13A	109.5	O5—C22—H22C	109.5
C11—C13—H13B	109.5	H22A—C22—H22B	109.5
C11—C13—H13C	109.5	H22A—C22—H22C	109.5
H13A—C13—H13B	109.5	H22B—C22—H22C	109.5
H13A—C13—H13C	109.5		

O1—C6—C7—C2	177.15 (14)	C6—O1—C17—O2	−2.0 (3)
O1—C6—C7—C8	−2.1 (3)	C6—O1—C17—C18	−179.77 (15)
O1—C17—C18—C19	−177.50 (17)	C6—C7—C8—C1	179.74 (19)
O2—C17—C18—C19	4.8 (3)	C6—C7—C8—C9	7.9 (3)
N1—C1—C8—C7	−0.1 (2)	C7—C2—C3—C4	0.7 (3)
N1—C1—C8—C9	172.23 (16)	C7—C8—C9—C10	90.2 (2)
N1—C2—C3—C4	179.42 (19)	C8—C9—C10—N2	179.92 (14)
N1—C2—C7—C6	179.94 (15)	C10—N2—C11—C13	−63.01 (19)
N1—C2—C7—C8	−0.61 (19)	C10—N2—C11—C12	63.1 (2)
C1—N1—C2—C3	−178.34 (19)	C10—N2—C14—C15	−178.31 (17)
C1—N1—C2—C7	0.5 (2)	C10—N2—C14—C16	58.79 (19)
C1—C8—C9—C10	−80.3 (2)	C11—N2—C10—C9	125.26 (16)
C2—N1—C1—C8	−0.3 (2)	C11—N2—C14—C15	−51.2 (2)
C2—C3—C4—C5	0.3 (3)	C11—N2—C14—C16	−174.13 (16)
C2—C7—C8—C1	0.45 (19)	C14—N2—C10—C9	−106.70 (17)
C2—C7—C8—C9	−171.42 (16)	C14—N2—C11—C13	170.27 (16)
C3—C2—C7—C6	−1.1 (2)	C14—N2—C11—C12	−63.6 (2)
C3—C2—C7—C8	178.39 (16)	C17—O1—C6—C5	−89.2 (2)
C3—C4—C5—C6	−0.8 (3)	C17—O1—C6—C7	94.06 (19)
C4—C5—C6—O1	−176.20 (17)	C17—C18—C19—C20	179.16 (18)
C4—C5—C6—C7	0.4 (3)	C18—C19—C20—C21	173.75 (19)
C5—C6—C7—C2	0.5 (2)	C19—C20—C21—O3	−6.2 (3)
C5—C6—C7—C8	−178.76 (19)	C19—C20—C21—O4	173.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*A*···O4	1.00 (1)	1.83 (2)	2.748 (2)	151 (3)
N2—H2···O4^{i}	0.90 (1)	1.81 (1)	2.7154 (19)	177 (2)
N1—H1···O3^{ii}	0.86 (1)	1.99 (1)	2.773 (2)	151 (2)

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

5-[(3-{2-[Bis(propan-2-yl)azaniumyl]ethyl}-1*H*-indol-4-yl)oxy]-5-oxopentanoate ethanol monosolvate (II)

Crystal data

$C_{21}H_{30}N_2O_4{\cdot}C_2H_6O$
M_r = 420.54
Monoclinic, $P2_1/c$
a = 8.0087 (12) Å
b = 13.7968 (17) Å
c = 21.878 (3) Å
β = 90.749 (4)°
V = 2417.2 (5) Å^3
Z = 4
$F(000)$ = 912
D_x = 1.156 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7678 reflections
θ = 2.5–24.9°

μ = 0.08 mm^{-1}
T = 297 K

Block, colourless
0.30 × 0.27 × 0.22 mm

Data collection

Bruker D8 Venture CMOS diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2021)
T_{min} = 0.692, T_{max} = 0.745
37412 measured reflections

4461 independent reflections
3038 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 25.4°, θ_{min} = 2.5°
h = −9→9
k = −16→16
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.176
S = 1.04
4461 reflections
313 parameters
46 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0772P)^2 + 1.2353P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.2092 (2)	0.36531 (11)	0.30305 (7)	0.0553 (5)	
O2	0.2584 (3)	0.52278 (14)	0.28903 (8)	0.0832 (7)	
O3	0.3563 (3)	0.73209 (16)	0.48029 (8)	0.0872 (7)	
O4	0.2945 (3)	0.67092 (14)	0.56965 (8)	0.0787 (7)	
N1	0.4952 (3)	0.27474 (16)	0.12915 (9)	0.0595 (6)	
N2	0.6135 (3)	0.16000 (15)	0.37926 (9)	0.0508 (5)	
C1	0.5975 (4)	0.26944 (19)	0.17914 (11)	0.0583 (7)	
H1A	0.708687	0.250056	0.178280	0.070*	
C2	0.3413 (4)	0.30442 (17)	0.14673 (10)	0.0513 (6)	
C3	0.1947 (4)	0.31751 (19)	0.11340 (12)	0.0630 (7)	
H3	0.191217	0.307090	0.071410	0.076*	
C4	0.0551 (4)	0.3462 (2)	0.14408 (13)	0.0701 (8)	
H4	−0.044828	0.354362	0.122584	0.084*	
C5	0.0599 (4)	0.3635 (2)	0.20711 (12)	0.0628 (7)	
H5	−0.035912	0.383807	0.226972	0.075*	
C6	0.2045 (3)	0.35080 (17)	0.23928 (10)	0.0504 (6)	
C7	0.3503 (3)	0.31992 (15)	0.21087 (10)	0.0461 (6)	
C8	0.5159 (3)	0.29619 (16)	0.23053 (10)	0.0492 (6)	
C9	0.5832 (3)	0.28496 (17)	0.29440 (11)	0.0532 (6)	
H9A	0.528225	0.330228	0.321445	0.064*	

H9B	0.702044	0.298597	0.295389	0.064*	
C10	0.5518 (3)	0.18157 (17)	0.31541 (10)	0.0513 (6)	
H10A	0.432721	0.168963	0.313271	0.062*	
H10B	0.605720	0.137478	0.287333	0.062*	
C11	0.7716 (4)	0.0990 (2)	0.37851 (13)	0.0689 (8)	
H11	0.745795	0.037954	0.357516	0.083*	
C12	0.8324 (5)	0.0750 (3)	0.44274 (16)	0.0990 (12)	
H12A	0.753581	0.032798	0.461969	0.148*	
H12B	0.843001	0.133711	0.466050	0.148*	
H12C	0.938971	0.043428	0.440839	0.148*	
C13	0.9059 (4)	0.1506 (3)	0.34291 (17)	0.0903 (11)	
H13A	0.866622	0.162243	0.301912	0.135*	
H13B	1.004393	0.111058	0.341935	0.135*	
H13C	0.931685	0.211311	0.362305	0.135*	
C14	0.4773 (4)	0.11792 (19)	0.41877 (12)	0.0615 (7)	
H14	0.526311	0.106134	0.459368	0.074*	
C15	0.4148 (5)	0.0211 (2)	0.39449 (17)	0.0947 (12)	
H15A	0.505966	−0.023937	0.393021	0.142*	
H15B	0.368656	0.029758	0.354130	0.142*	
H15C	0.330151	−0.003624	0.420973	0.142*	
C16	0.3377 (4)	0.1903 (2)	0.42646 (14)	0.0753 (9)	
H16A	0.255515	0.164077	0.453397	0.113*	
H16B	0.286909	0.203677	0.387375	0.113*	
H16C	0.381923	0.249228	0.443505	0.113*	
C17	0.2386 (3)	0.45697 (18)	0.32284 (11)	0.0538 (6)	
C18	0.2450 (4)	0.46119 (18)	0.39101 (11)	0.0596 (7)	
H18A	0.336436	0.420965	0.405661	0.072*	
H18B	0.142255	0.434400	0.406826	0.072*	
C19	0.2678 (4)	0.56242 (19)	0.41582 (11)	0.0625 (7)	
H19A	0.373719	0.587837	0.402005	0.075*	
H19B	0.179984	0.603603	0.399319	0.075*	
C20	0.2642 (4)	0.5668 (2)	0.48422 (11)	0.0690 (8)	
H20A	0.341419	0.518749	0.500346	0.083*	
H20B	0.153216	0.548829	0.497359	0.083*	
C21	0.3081 (4)	0.6643 (2)	0.51231 (11)	0.0627 (7)	
H2	0.640 (3)	0.217 (2)	0.3959 (12)	0.064 (8)*	
H1	0.526 (3)	0.261 (2)	0.0929 (6)	0.068 (8)*	
O5	−0.1278 (12)	0.4643 (7)	0.3667 (5)	0.123 (3)	0.531 (11)
H5A	−0.165922	0.438097	0.397138	0.184*	0.531 (11)
C22	−0.2489 (13)	0.5309 (7)	0.3417 (5)	0.106 (3)	0.531 (11)
H22A	−0.281815	0.577081	0.372735	0.127*	0.531 (11)
H22B	−0.347635	0.495888	0.328047	0.127*	0.531 (11)
C23	−0.1720 (13)	0.5827 (6)	0.2891 (3)	0.106 (3)	0.531 (11)
H23A	−0.251356	0.627541	0.271841	0.159*	0.531 (11)
H23B	−0.074882	0.617423	0.303038	0.159*	0.531 (11)
H23C	−0.140469	0.536518	0.258509	0.159*	0.531 (11)
O5A	−0.1337 (9)	0.4474 (4)	0.3501 (3)	0.0655 (18)	0.469 (11)
H5AA	−0.198660	0.410977	0.367474	0.098*	0.469 (11)

C22A	−0.1963 (16)	0.5437 (6)	0.3520 (4)	0.098 (3)	0.469 (11)
H22C	−0.106576	0.590542	0.356016	0.117*	0.469 (11)
H22D	−0.273691	0.552244	0.385271	0.117*	0.469 (11)
C23A	−0.2814 (13)	0.5518 (7)	0.2923 (4)	0.097 (3)	0.469 (11)
H23D	−0.329689	0.615146	0.288185	0.146*	0.469 (11)
H23E	−0.202252	0.541673	0.260303	0.146*	0.469 (11)
H23F	−0.367862	0.503720	0.289294	0.146*	0.469 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0813 (13)	0.0451 (9)	0.0399 (9)	−0.0016 (8)	0.0149 (8)	−0.0089 (7)
O2	0.142 (2)	0.0566 (12)	0.0510 (11)	−0.0251 (12)	0.0040 (12)	0.0006 (9)
O3	0.142 (2)	0.0749 (13)	0.0456 (10)	−0.0433 (13)	0.0284 (11)	−0.0115 (10)
O4	0.1303 (19)	0.0686 (12)	0.0376 (9)	−0.0367 (12)	0.0170 (10)	−0.0126 (8)
N1	0.0858 (17)	0.0567 (13)	0.0364 (11)	0.0033 (11)	0.0155 (11)	−0.0061 (9)
N2	0.0669 (14)	0.0427 (11)	0.0428 (11)	−0.0010 (10)	0.0009 (9)	−0.0050 (9)
C1	0.0704 (18)	0.0556 (15)	0.0491 (14)	0.0020 (13)	0.0113 (12)	−0.0018 (11)
C2	0.0757 (18)	0.0403 (12)	0.0382 (12)	−0.0019 (12)	0.0086 (12)	−0.0034 (10)
C3	0.090 (2)	0.0592 (16)	0.0391 (13)	−0.0040 (15)	−0.0046 (14)	−0.0045 (11)
C4	0.075 (2)	0.0740 (19)	0.0604 (17)	−0.0038 (16)	−0.0109 (15)	−0.0013 (14)
C5	0.0634 (18)	0.0629 (17)	0.0621 (16)	0.0023 (13)	0.0069 (14)	−0.0077 (13)
C6	0.0693 (17)	0.0428 (13)	0.0392 (12)	−0.0041 (11)	0.0078 (11)	−0.0071 (10)
C7	0.0662 (16)	0.0360 (11)	0.0360 (11)	−0.0054 (10)	0.0050 (10)	−0.0042 (9)
C8	0.0650 (16)	0.0410 (12)	0.0417 (12)	−0.0040 (11)	0.0050 (11)	−0.0024 (10)
C9	0.0671 (17)	0.0480 (14)	0.0444 (13)	−0.0043 (12)	0.0013 (11)	−0.0063 (10)
C10	0.0649 (16)	0.0482 (14)	0.0407 (12)	−0.0041 (12)	−0.0010 (11)	−0.0045 (10)
C11	0.078 (2)	0.0663 (18)	0.0619 (16)	0.0174 (15)	−0.0067 (14)	−0.0074 (14)
C12	0.103 (3)	0.113 (3)	0.080 (2)	0.033 (2)	−0.021 (2)	0.003 (2)
C13	0.066 (2)	0.112 (3)	0.094 (2)	0.0162 (19)	0.0070 (18)	−0.011 (2)
C14	0.081 (2)	0.0516 (14)	0.0520 (14)	−0.0085 (14)	0.0075 (13)	0.0042 (12)
C15	0.126 (3)	0.0562 (18)	0.102 (3)	−0.0246 (19)	0.022 (2)	0.0006 (17)
C16	0.078 (2)	0.076 (2)	0.0728 (19)	−0.0069 (16)	0.0166 (16)	−0.0036 (15)
C17	0.0651 (17)	0.0494 (14)	0.0470 (13)	−0.0051 (12)	0.0068 (11)	−0.0079 (11)
C18	0.0809 (19)	0.0515 (14)	0.0466 (14)	−0.0031 (13)	0.0096 (13)	−0.0091 (11)
C19	0.083 (2)	0.0556 (15)	0.0487 (14)	−0.0081 (14)	0.0050 (13)	−0.0121 (12)
C20	0.098 (2)	0.0628 (17)	0.0465 (14)	−0.0181 (15)	0.0105 (14)	−0.0130 (12)
C21	0.084 (2)	0.0624 (16)	0.0420 (13)	−0.0190 (14)	0.0143 (13)	−0.0109 (12)
O5	0.125 (4)	0.120 (4)	0.123 (4)	0.0016 (19)	0.0008 (19)	0.0101 (19)
C22	0.106 (3)	0.105 (3)	0.107 (3)	0.0010 (10)	0.0003 (10)	−0.0001 (10)
C23	0.107 (3)	0.104 (3)	0.106 (3)	0.0061 (19)	0.0028 (19)	0.0021 (19)
O5A	0.070 (2)	0.060 (2)	0.067 (2)	−0.0011 (15)	0.0075 (16)	0.0087 (16)
C22A	0.098 (3)	0.096 (3)	0.099 (3)	0.0021 (10)	0.0001 (10)	0.0004 (10)
C23A	0.098 (4)	0.094 (3)	0.100 (3)	0.0024 (19)	−0.0061 (19)	0.0016 (19)

Geometric parameters (Å, º)

O1—C6	1.409 (3)	C13—H13B	0.9600
O1—C17	1.356 (3)	C13—H13C	0.9600
O2—C17	1.183 (3)	C14—H14	0.9800
O3—C21	1.234 (3)	C14—C15	1.520 (4)
O4—C21	1.264 (3)	C14—C16	1.510 (4)
N1—C1	1.360 (4)	C15—H15A	0.9600
N1—C2	1.359 (3)	C15—H15B	0.9600
N1—H1	0.853 (10)	C15—H15C	0.9600
N2—C10	1.505 (3)	C16—H16A	0.9600
N2—C11	1.521 (4)	C16—H16B	0.9600
N2—C14	1.517 (3)	C16—H16C	0.9600
N2—H2	0.90 (3)	C17—C18	1.493 (3)
C1—H1A	0.9300	C18—H18A	0.9700
C1—C8	1.359 (3)	C18—H18B	0.9700
C2—C3	1.386 (4)	C18—C19	1.509 (3)
C2—C7	1.420 (3)	C19—H19A	0.9700
C3—H3	0.9300	C19—H19B	0.9700
C3—C4	1.370 (4)	C19—C20	1.498 (3)
C4—H4	0.9300	C20—H20A	0.9700
C4—C5	1.400 (4)	C20—H20B	0.9700
C5—H5	0.9300	C20—C21	1.518 (4)
C5—C6	1.358 (4)	O5—H5A	0.8200
C6—C7	1.397 (3)	O5—C22	1.439 (8)
C7—C8	1.427 (4)	C22—H22A	0.9700
C8—C9	1.499 (3)	C22—H22B	0.9700
C9—H9A	0.9700	C22—C23	1.496 (8)
C9—H9B	0.9700	C23—H23A	0.9600
C9—C10	1.521 (3)	C23—H23B	0.9600
C10—H10A	0.9700	C23—H23C	0.9600
C10—H10B	0.9700	O5A—H5AA	0.8200
C11—H11	0.9800	O5A—C22A	1.420 (8)
C11—C12	1.517 (4)	C22A—H22C	0.9700
C11—C13	1.514 (5)	C22A—H22D	0.9700
C12—H12A	0.9600	C22A—C23A	1.470 (8)
C12—H12B	0.9600	C23A—H23D	0.9600
C12—H12C	0.9600	C23A—H23E	0.9600
C13—H13A	0.9600	C23A—H23F	0.9600
C17—O1—C6	116.79 (18)	C15—C14—H14	107.3
C1—N1—H1	124 (2)	C16—C14—N2	110.5 (2)
C2—N1—C1	109.1 (2)	C16—C14—H14	107.3
C2—N1—H1	127 (2)	C16—C14—C15	112.3 (3)
C10—N2—C11	111.28 (19)	C14—C15—H15A	109.5
C10—N2—C14	112.0 (2)	C14—C15—H15B	109.5
C10—N2—H2	106.1 (17)	C14—C15—H15C	109.5
C11—N2—H2	107.4 (18)	H15A—C15—H15B	109.5

C14—N2—C11	113.6 (2)	H15A—C15—H15C	109.5
C14—N2—H2	106.0 (17)	H15B—C15—H15C	109.5
N1—C1—H1A	124.5	C14—C16—H16A	109.5
C8—C1—N1	111.1 (3)	C14—C16—H16B	109.5
C8—C1—H1A	124.5	C14—C16—H16C	109.5
N1—C2—C3	131.0 (2)	H16A—C16—H16B	109.5
N1—C2—C7	106.9 (2)	H16A—C16—H16C	109.5
C3—C2—C7	122.1 (2)	H16B—C16—H16C	109.5
C2—C3—H3	120.9	O1—C17—C18	111.0 (2)
C4—C3—C2	118.1 (2)	O2—C17—O1	122.7 (2)
C4—C3—H3	120.9	O2—C17—C18	126.3 (2)
C3—C4—H4	119.3	C17—C18—H18A	108.9
C3—C4—C5	121.3 (3)	C17—C18—H18B	108.9
C5—C4—H4	119.3	C17—C18—C19	113.5 (2)
C4—C5—H5	120.0	H18A—C18—H18B	107.7
C6—C5—C4	120.0 (3)	C19—C18—H18A	108.9
C6—C5—H5	120.0	C19—C18—H18B	108.9
C5—C6—O1	120.4 (2)	C18—C19—H19A	109.0
C5—C6—C7	121.4 (2)	C18—C19—H19B	109.0
C7—C6—O1	118.2 (2)	H19A—C19—H19B	107.8
C2—C7—C8	107.4 (2)	C20—C19—C18	113.1 (2)
C6—C7—C2	117.0 (2)	C20—C19—H19A	109.0
C6—C7—C8	135.6 (2)	C20—C19—H19B	109.0
C1—C8—C7	105.6 (2)	C19—C20—H20A	108.4
C1—C8—C9	124.9 (2)	C19—C20—H20B	108.4
C7—C8—C9	128.8 (2)	C19—C20—C21	115.7 (2)
C8—C9—H9A	110.0	H20A—C20—H20B	107.4
C8—C9—H9B	110.0	C21—C20—H20A	108.4
C8—C9—C10	108.65 (19)	C21—C20—H20B	108.4
H9A—C9—H9B	108.3	O3—C21—O4	122.7 (2)
C10—C9—H9A	110.0	O3—C21—C20	121.0 (2)
C10—C9—H9B	110.0	O4—C21—C20	116.3 (2)
N2—C10—C9	114.35 (19)	C22—O5—H5A	109.5
N2—C10—H10A	108.7	O5—C22—H22A	110.0
N2—C10—H10B	108.7	O5—C22—H22B	110.0
C9—C10—H10A	108.7	O5—C22—C23	108.4 (8)
C9—C10—H10B	108.7	H22A—C22—H22B	108.4
H10A—C10—H10B	107.6	C23—C22—H22A	110.0
N2—C11—H11	108.1	C23—C22—H22B	110.0
C12—C11—N2	111.6 (2)	C22—C23—H23A	109.5
C12—C11—H11	108.1	C22—C23—H23B	109.5
C13—C11—N2	110.0 (2)	C22—C23—H23C	109.5
C13—C11—H11	108.1	H23A—C23—H23B	109.5
C13—C11—C12	111.0 (3)	H23A—C23—H23C	109.5
C11—C12—H12A	109.5	H23B—C23—H23C	109.5
C11—C12—H12B	109.5	C22A—O5A—H5AA	109.5
C11—C12—H12C	109.5	O5A—C22A—H22C	111.4
H12A—C12—H12B	109.5	O5A—C22A—H22D	111.4

H12A—C12—H12C	109.5	O5A—C22A—C23A	101.9 (7)
H12B—C12—H12C	109.5	H22C—C22A—H22D	109.3
C11—C13—H13A	109.5	C23A—C22A—H22C	111.4
C11—C13—H13B	109.5	C23A—C22A—H22D	111.4
C11—C13—H13C	109.5	C22A—C23A—H23D	109.5
H13A—C13—H13B	109.5	C22A—C23A—H23E	109.5
H13A—C13—H13C	109.5	C22A—C23A—H23F	109.5
H13B—C13—H13C	109.5	H23D—C23A—H23E	109.5
N2—C14—H14	107.3	H23D—C23A—H23F	109.5
N2—C14—C15	111.9 (2)	H23E—C23A—H23F	109.5
O1—C6—C7—C2	179.04 (19)	C6—O1—C17—O2	0.6 (4)
O1—C6—C7—C8	0.8 (4)	C6—O1—C17—C18	−178.4 (2)
O1—C17—C18—C19	−176.7 (2)	C6—C7—C8—C1	179.0 (3)
O2—C17—C18—C19	4.3 (5)	C6—C7—C8—C9	8.9 (4)
N1—C1—C8—C7	0.0 (3)	C7—C2—C3—C4	−0.1 (4)
N1—C1—C8—C9	170.6 (2)	C7—C8—C9—C10	86.1 (3)
N1—C2—C3—C4	178.3 (3)	C8—C9—C10—N2	180.0 (2)
N1—C2—C7—C6	−179.8 (2)	C10—N2—C11—C12	−178.7 (3)
N1—C2—C7—C8	−1.1 (3)	C10—N2—C11—C13	57.7 (3)
C1—N1—C2—C3	−177.5 (3)	C10—N2—C14—C15	62.8 (3)
C1—N1—C2—C7	1.1 (3)	C10—N2—C14—C16	−63.1 (3)
C1—C8—C9—C10	−82.3 (3)	C11—N2—C10—C9	−105.4 (3)
C2—N1—C1—C8	−0.7 (3)	C11—N2—C14—C15	−64.3 (3)
C2—C3—C4—C5	1.0 (4)	C11—N2—C14—C16	169.8 (2)
C2—C7—C8—C1	0.7 (3)	C14—N2—C10—C9	126.3 (2)
C2—C7—C8—C9	−169.4 (2)	C14—N2—C11—C12	−51.2 (3)
C3—C2—C7—C6	−1.0 (3)	C14—N2—C11—C13	−174.8 (2)
C3—C2—C7—C8	177.7 (2)	C17—O1—C6—C5	−87.0 (3)
C3—C4—C5—C6	−0.8 (4)	C17—O1—C6—C7	95.2 (3)
C4—C5—C6—O1	−178.1 (2)	C17—C18—C19—C20	176.7 (3)
C4—C5—C6—C7	−0.4 (4)	C18—C19—C20—C21	172.6 (3)
C5—C6—C7—C2	1.2 (3)	C19—C20—C21—O3	−5.6 (5)
C5—C6—C7—C8	−177.0 (3)	C19—C20—C21—O4	175.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O4^{i}	0.90 (3)	1.79 (3)	2.686 (3)	177 (3)
N1—H1···O3^{ii}	0.85 (1)	1.91 (1)	2.751 (3)	167 (3)
O5—H5*A*···O4^{iii}	0.82	1.97	2.692 (10)	147
O5*A*—H5*AA*···O4^{iii}	0.82	1.95	2.732 (6)	160

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