Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal water as the molecular glue for obtaining different co-crystal ratios: the case of gallic acid tris-caffeine hexahydrate

L. Vella-Zarb and U. Baisch*

University of Malta, Msida, MSD 2080, Malta. *Correspondence e-mail: ulrich.baisch@um.edu.mt

Received 9 March 2018
Accepted 18 March 2018

Edited by A. J. Lough, University of Toronto, Canada

Keywords: crystal structure; organic co-crystal; hydrate; gallic acid; caffeine.

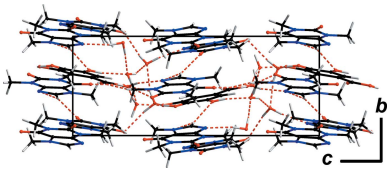
CCDC reference: 1830592

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

The crystal structure of the hexahydrate co-crystal of gallic acid and caffeine, $C_7H_6O_5\cdot3C_8H_{10}N_4O_2\cdot6H_2O$ or **GAL3CAF·6H$_2$O**, is a remarkable example of the importance of hydrate water acting as structural glue to facilitate the crystallization of two components of different stoichiometries and thus to compensate an imbalance of hydrogen-bond donors and acceptors. The water molecules provide the additional hydrogen bonds required to form a crystalline solid. Whereas the majority of hydrogen bonds forming the intermolecular network between gallic acid and caffeine are formed by crystal water, only one direct classical hydrogen bond between two molecules is formed between the carboxylic oxygen of gallic acid and the carbonyl oxygen of caffeine with $d(D\cdots A)$ = 2.672 (2) Å. All other hydrogen bonds either involve crystal water or utilize protonated carbon atoms as donors.

1. Chemical context

Gallic acid and its derivatives are widely known compounds in the pharmaceutical and chemical industry (Nayeem *et al.*, 2016; Clarke *et al.*, 2011). One such example is the dietary polyphenol found in *Choerospondiatis fructus*, a Mongolian medicinal herb used to treat conditions such as angina pectoris (Zhao *et al.*, 2007). Lately, it has gained a lot of attention as a versatile component in crystal enineering, in particular with regards to co-crystallization and hydratation. Gallic acid could represent an entire microcosm of the special challenges and opportunities afforded by hydrates (Clarke *et al.*, 2011) as it contains two of the most ubiquitous functional groups present in APIs: carboxylic acids and phenols. As part of a series of co-crystallization experiments in which both caffeine and gallic acid were used as coformers, single crystals of hydrated gallic acid and caffeine **GAL3CAF·6H$_2$O** in the ratio gallic acid:-caffeine:water of 1:3:6 were obtained and characterized by single-crystal X-ray diffraction. The crystal structure is reported herein and compared to the different hydrated forms of this co-crystal **GALCAF·0.5H$_2$O** reported elsewhere (Clarke *et al.*, 2010). The crystal structures differ greatly because of the different stoichiometry of the coformers. The different number of water molecules is necessary to act as structural glue, thereby facilitating crystallization.



2. Structural commentary

The asymmetric unit of the co-crystal **GAL3CAF·6H$_2$O** consists of three independent caffeine molecules and one

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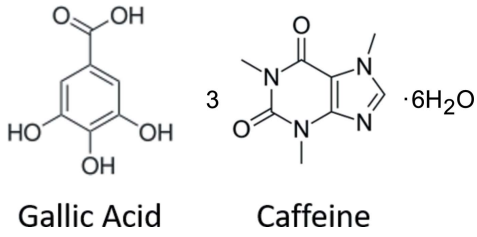
gallic acid molecule as well as six hydrate water molecules. Gallic acid can be described as a thrice-substituted benzoic acid with hydroxyl groups in both the *meta* and *para* positons. Caffeine consists of a purine backbone with carbonyl substituents at positions 2 and 6 (C26, C28, C46, C48, C66, C68) and methyl groups connected to three out of four nitrogen atoms (Fig. 1).



Bond distances of the aromatic rings and substituents of both types of molecules lie within the expected ranges and exhibit the usual lengths for aromatic, double or single homo- or heteroatomic bonds. Only one nitrogen atom on each of the three caffeine molecules can act as a hydrogen-bond acceptor (N23, N43, N63) with the protonated carbon in the five-membered ring (C22, C42, C62) acting as a weak hydrogen-bond donor. Every molecule exhibits weak intramolecular interactions (Steiner, 2002). Whereas gallic acid forms intermoleclar bonds between two adjacent hydroxyl substituents [O11—H11···O10, $d(D\cdots A)$ = 2.709 (2) Å], the caffeine molecules form weak interactions between two of three methyl carbons and two carbonyl oxygen or backbone nitrogen atoms, namely C31, C33, C51, C53, C71, C73 as well as O32, N43, O54, O72, and O74. Distances range between 2.71 and 2.95 Å. In comparison, the corresponding intramolecular hydrogen-bonding interactions in the published **GALCAF·0.5H$_2$O** structure reported in the Cambridge Structural Database (Groom *et al.*, 2016) with code MUPNOB (Clarke *et al.*, 2010) have distances of $d(D\cdots A)_{\text{gallic acid}}$ = 2.743 (2), 2.712 (2) Å and $d(D\cdots A)_{\text{caffeine}}$ of 2.78–2.71 Å.

3. Supramolecular features

As a result of the limited number of hydrogen-bond donors and acceptors in both gallic acid and caffeine, the packing strongly depends on (i) the concentration of each of the components in solution as well as (ii) other experimental conditions such as other components in solution, temperature, pressure, *etc*. In fact, there is a large difference in the way both molecules pack in the crystal lattice.

The crystal structure of **GALCAF·0.5H$_2$O** (Clarke *et al.*, 2010) has a 1:1:0.5 ratio of gallic acid, caffeine and water molecules. Both molecules form hydrogen-bonded tapes that are built by COO—H···N and O—H···O interactions [O···N = 2.705 (2) Å and O···O = 2.703 (2) and 2.750 (2) Å] formed between the hydoxyl substituents on gallic acid molecules and the carbonyl moieties of adjacent caffeine molecules. These tapes are then cross-linked by water molecules that hydrogen-bond with the third hydroxyl group in each gallic acid molecule [O···O = 2.857 (1) Å]. The water molecules facilitate the formation of bilayers that stack in an *ABAB* manner sustained by π–π interactions. The distances between these layers of molecules can be calculated from the distances between the centroids of the aromatic rings of the two molecules and range from 3.3742 (14) to 4.3402 (14) Å. The ratio between classical hydrogen-bond donors and acceptors is 4:4 (four donors and one acceptor on the gallic acid molecule and three acceptors on the caffeine molecule).

The different balance of gallic acid and caffeine in **GAL3CAF·6H$_2$O** affects the donor/acceptor ratio significantly. There are still only four classical hydrogen-bond

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O32^{i}	0.85 (3)	1.86 (3)	2.672 (2)	161 (3)
O10—H10···O104	0.86 (4)	1.79 (4)	2.643 (2)	174 (4)
O11—H11···O10	0.83 (3)	2.32 (4)	2.709 (2)	109 (3)
O11—H11···O102	0.83 (4)	1.88 (4)	2.680 (2)	161 (3)
O12—H12···O100^{i}	0.86 (3)	1.86 (3)	2.702 (2)	166 (3)
O100—H10*C*···O105	0.83 (4)	2.07 (4)	2.851 (3)	157 (4)
O100—H10*D*···O10	0.82 (4)	2.74 (3)	3.286 (2)	126 (3)
O100—H10*D*···O102	0.82 (4)	2.02 (4)	2.798 (3)	158 (3)
O101—H10*I*···N43^{ii}	0.94 (4)	1.97 (4)	2.898 (3)	170 (3)
O101—H10*J*···O100	0.88 (4)	2.09 (4)	2.960 (3)	170 (4)
O102—H10*A*···O101^{iii}	0.89 (4)	1.87 (4)	2.754 (3)	169 (4)
O102—H10*B*···O103	0.85 (4)	1.85 (4)	2.691 (3)	173 (3)
O103—H10*K*···O34^{iv}	0.78 (5)	2.06 (5)	2.832 (3)	168 (5)
O103—H10*L*···N23^{v}	0.84 (5)	1.99 (5)	2.823 (3)	168 (4)
O104—H10*G*···O1^{vi}	0.91 (3)	2.50 (3)	3.011 (2)	116 (2)
O104—H10*G*···O105	0.91 (3)	2.03 (3)	2.857 (3)	151 (3)
O104—H10*H*···O74^{vi}	0.93 (5)	2.00 (5)	2.924 (2)	171 (4)
O105—H10*E*···O74^{ii}	0.86 (5)	2.11 (5)	2.952 (2)	169 (4)
O105—H10*F*···N63	0.88 (4)	1.92 (4)	2.798 (2)	173 (4)
C22—H22···O72^{vii}	0.97 (3)	2.38 (3)	3.227 (3)	146 (2)
C30—H30*C*···O12^{vii}	0.98	2.71	3.247 (3)	115
C31—H31*C*···O2^{vi}	0.98	2.40	3.326 (3)	158
C42—H42···O12^{viii}	0.92 (3)	2.45 (3)	3.254 (3)	146 (3)
C42—H42···O72	0.92 (3)	2.66 (3)	3.136 (3)	113 (2)
C62—H62···O52^{vii}	0.96 (3)	2.24 (3)	3.119 (3)	151 (3)

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

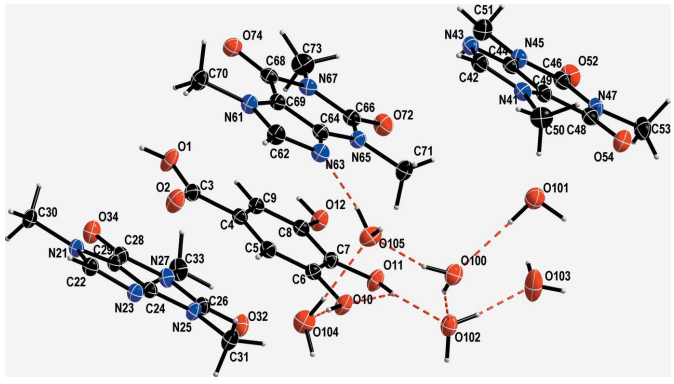


Figure 1
Molecular structure of the **GAL3CAF·6H$_2$O** showing the labelling scheme and displacement ellipsoids drawn at 50% probability level.

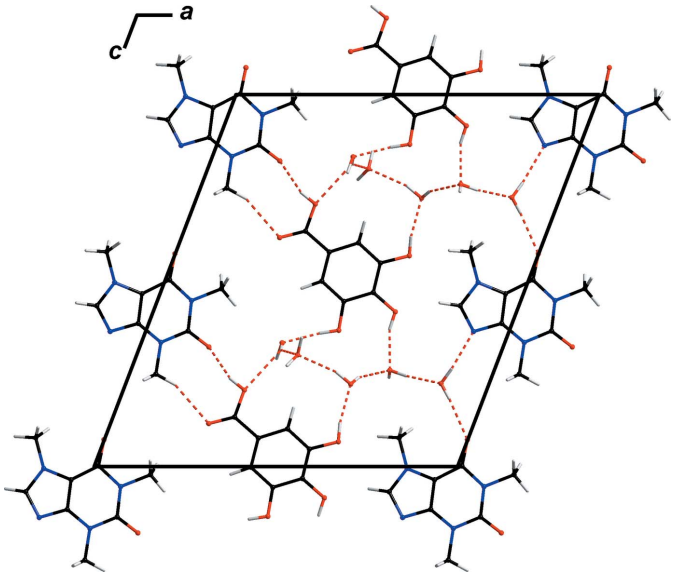


Figure 2
Crystal packing of **GAL3CAF·6H$_2$O** viewed along *b*. Hydrogen-bonding interactions (Table 1) are shown as red dashed lines.

donors deriving from the hydroxyl groups on gallic acid, but ten hydrogen-bond acceptors (three on each caffeine and one on gallic acid). This discrepency is equilibrated by inclusion of additional solvent water molecules into the crystal structure. These act as structural glue enabling crystallization in different stoichiometries and thus, compensating for the above imbalance. The water molecules provide the additional hydrogen bonds required to form a crystalline solid. Thus, the majority of hydrogen bonds forming the intermolecular network between gallic acid and caffeine are formed by crystal water (Table 1) with $d(D\cdots A)$ ranging from 2.643 (2) to 3.011 (2) Å. Direct classical hydrogen bonding between non-carbon atoms can only be observed between the carboxylic oxygen of gallic acid (O1) and the carbonyl oxygen of caffeine (O32) with $d(D\cdots A)$ = 2.672 (2) Å (Fig. 2). Additionally, there is a significant number of weak C—H···O interactions present between gallic acid molecules and caffeine molecules

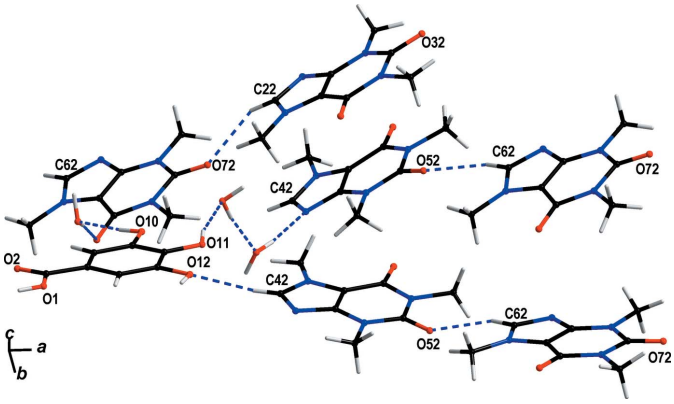


Figure 3
Crystal packing of **GAL3CAF·6H$_2$O**. Hydrogen-bonding interactions (Table 1) are shown as blue dashed lines.

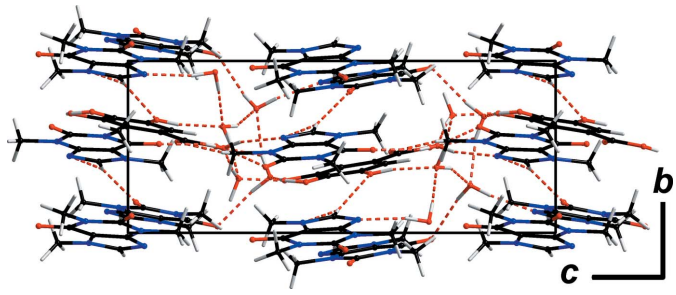


Figure 4
Crystal packing of **GAL3CAF·6H$_2$O** viewed along *a*. Hydrogen-bonding interactions are shown as red dashed lines.

and between caffeine molecules themselves (Table 1). One of these interactions is between carboxylic acid and a carbonyl oxygen *via* hydrogen bonding that is almost parallel to an interaction between the carbonyl oxygen (O2) of the carboxylic group in gallic acid with the adjacent proton of a caffeine methyl substituent (C31) $d(D\cdots A)$ 3.326 (3) Å. Another C—H···O interaction is notable as it forms a linear chain connecting all caffeine molecules to each other (Fig. 3). These are formed between the only protonated carbon atom in the purine backbone (C22—H22, C42—H42, C62—H62) and the carbonyl oxygen of the next caffeine molecule (O32, O52, O72) with donor–acceptor distances ranging from 3.119 (3) to 3.227 (3) Å. O12 links gallic acid molecules to these chains *via* additional weak C—H···O interactions to C30 [$d(D\cdots A)$ 3.247 (3) Å] and C42 [$d(D\cdots A)$ = 3.254 (3) Å]. A comparable interaction between the protonated carbon of the purine ring does not exist in the **GALCAF·0.5H$_2$O** structure.

The crystal structure of **GAL3CAF·6H$_2$O** can be described as having two types of molecular layers connected *via* hydrogen-bonding interactions with solvent water molecules. Layers consisting solely of caffeine molecules are stacked alternately with layers composed of caffeine and gallic acid molecules (Fig. 4). The distances between the centroids of the aromatic rings are within the significance range at 3.231 (13) and 4.5028 (13) Å. Thus π-stacking of the aromatic rings is both stronger and weaker in places.

The discussed crystal structure provides a good representation of the large impact of weak C—H···O interactions and of how solvent molecules can play a crucial role in the formation of crystal structures. All our attempts at obtaining a solventless co-crystal with the same stoichiometry have failed so far.

4. Synthesis and crystallization

The crystals were obtained as a by-product in a reaction aiming for the synthesis of a lanthanide salt. Gel crystallizations were carried out in order to slow the crystallization process down. This technique involves a piece of glassware that allows two solutions to diffuse through a (tetramethylorthosilicate) gel medium. The two sets of reagents then react when they eventually diffuse through the gel. Tetramethyl-

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_7H_6O_5{\cdot}3C_8H_{10}N_4O_2{\cdot}6H_2O$
M_r	860.81
Crystal system, space group	Monoclinic, *Pc*
Temperature (K)	150
a, *b*, *c* (Å)	16.5434 (3), 6.79456 (11), 18.1390 (4)
β (°)	110.865 (2)
V (Å^3)	1905.21 (7)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.12
Crystal size (mm)	0.66 × 0.37 × 0.06
Data collection	
Diffractometer	Rigaku Oxford Diffraction Xcalibur, Atlas, Gemini ultra
Absorption correction	Analytical (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.946, 0.994
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	38532, 9519, 8931
R_{int}	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.695
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.033, 0.086, 1.04
No. of reflections	9519
No. of parameters	645
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.23
Absolute structure	Flack *x* determined using 4000 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.1 (2)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg, 1999) and *OLEX2* (Dolomanov *et al.*, 2009).

orthosilicate gel (10%) was prepared freshly from 7 mL in 63 mL distilled water using Na_2CO_3 to make the gel approximately pH 8, and left to set overnight using U-tubes. Solutions were put into the two reservoirs: one contained caffeine (1 mmol, 0.2 g), while a solution containing an excess gallic acid and lanthanide was in the other.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Methyl H atoms were refined as riding (C—H = 0.98 Å with U_{iso}(H) = 1.5U_{eq}(C).

Funding information

Funding for this research was provided by: Seventh Framework Programme, FP7 People: Marie-Curie Actions (grant No. 256547 to U. Baisch).

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supporting information

Acta Cryst. (2018). E**74**, 559-562 [https://doi.org/10.1107/S2056989018004528]

Crystal water as the molecular glue for obtaining different co-crystal ratios: the case of gallic acid tris-caffeine hexahydrate

L. Vella-Zarb and U. Baisch

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2015); cell refinement: *CrysAlis PRO* (Rigaku OD, 2015); data reduction: *CrysAlis PRO* (Rigaku OD, 2015); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

Gallic Acid Tris-Caffeine hexahydrate

Crystal data

$C_7H_6O_5 \cdot 3C_8H_{10}N_4O_2 \cdot 6H_2O$
$M_r = 860.81$
Monoclinic, *Pc*
$a = 16.5434$ (3) Å
$b = 6.79456$ (11) Å
$c = 18.1390$ (4) Å
$\beta = 110.865$ (2)°
$V = 1905.21$ (7) Å^3
$Z = 2$
$F(000) = 908$
$D_x = 1.501$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 27647 reflections
$\theta = 3.0$–$29.6°$
$\mu = 0.12$ mm^{-1}
$T = 150$ K
Plate, clear colourless
0.66 × 0.37 × 0.06 mm

Data collection

Rigaku Oxford Diffraction Xcalibur, Atlas, Gemini ultra diffractometer
Detector resolution: 10.3968 pixels mm^{-1}
ω scans
Absorption correction: analytical (CrysAlisPro; Rigaku OD, 2015)
$T_{min} = 0.946$, $T_{max} = 0.994$
38532 measured reflections
9519 independent reflections
8931 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 29.6°$, $\theta_{min} = 3.0°$
$h = -21 \rightarrow 22$
$k = -9 \rightarrow 9$
$l = -25 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.086$
$S = 1.04$
9519 reflections
645 parameters
2 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 0.2608P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Absolute structure: Flack *x* determined using 4000 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.1 (2)

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}
O12	0.64601 (10)	0.3426 (2)	0.43298 (9)	0.0237 (3)
O74	0.39816 (11)	0.9622 (2)	0.29064 (9)	0.0249 (3)
O10	0.53021 (10)	0.3054 (3)	0.63346 (9)	0.0250 (3)
O72	0.66076 (10)	0.8227 (3)	0.48111 (10)	0.0279 (3)
O102	0.71299 (11)	0.3149 (3)	0.74098 (9)	0.0259 (3)
O11	0.66635 (9)	0.2955 (2)	0.58370 (9)	0.0241 (3)
O32	0.19013 (10)	0.3831 (2)	0.67800 (9)	0.0257 (3)
O34	−0.00090 (10)	0.4828 (2)	0.42738 (9)	0.0259 (3)
O54	1.06258 (11)	0.9604 (3)	0.70872 (10)	0.0289 (3)
O1	0.33315 (11)	0.4973 (3)	0.29402 (10)	0.0292 (4)
O100	0.61941 (12)	0.6181 (3)	0.77785 (10)	0.0266 (3)
O105	0.44257 (11)	0.7234 (3)	0.69929 (10)	0.0288 (3)
O52	1.14078 (10)	0.9016 (3)	0.49147 (10)	0.0300 (4)
O2	0.27018 (10)	0.4707 (3)	0.38411 (10)	0.0300 (4)
O104	0.38756 (11)	0.3238 (3)	0.66710 (11)	0.0312 (4)
O101	0.74791 (12)	0.9351 (3)	0.79622 (11)	0.0325 (4)
N27	0.09392 (11)	0.4384 (3)	0.55358 (11)	0.0200 (3)
N41	0.88046 (12)	1.0593 (3)	0.59097 (11)	0.0210 (3)
N23	−0.09763 (11)	0.5534 (3)	0.63666 (10)	0.0203 (3)
N67	0.52922 (11)	0.8905 (3)	0.38714 (11)	0.0201 (3)
N21	−0.13658 (11)	0.5677 (3)	0.50514 (10)	0.0187 (3)
N61	0.32462 (12)	0.9330 (3)	0.42452 (11)	0.0216 (4)
N63	0.41353 (12)	0.8759 (3)	0.54870 (11)	0.0221 (4)
O103	0.86742 (14)	0.4935 (4)	0.77642 (12)	0.0486 (6)
N45	1.00201 (12)	0.9877 (3)	0.46922 (11)	0.0213 (4)
N47	1.10076 (11)	0.9323 (3)	0.59887 (11)	0.0221 (4)
N43	0.85646 (11)	1.0770 (3)	0.46163 (11)	0.0223 (4)
N25	0.05332 (11)	0.4685 (3)	0.66583 (10)	0.0200 (3)
N65	0.54639 (11)	0.8526 (3)	0.52143 (10)	0.0193 (3)
C9	0.49273 (13)	0.4032 (3)	0.39853 (12)	0.0182 (4)
C28	0.01160 (13)	0.4814 (3)	0.49805 (12)	0.0185 (4)
C68	0.43992 (14)	0.9264 (3)	0.36060 (12)	0.0193 (4)
C64	0.45928 (13)	0.8801 (3)	0.49996 (12)	0.0188 (4)
C5	0.43204 (13)	0.3818 (3)	0.50144 (12)	0.0185 (4)
C22	−0.16156 (14)	0.5854 (3)	0.56754 (12)	0.0204 (4)
C4	0.42193 (13)	0.4139 (3)	0.42310 (12)	0.0180 (4)
C26	0.11628 (13)	0.4278 (3)	0.63532 (12)	0.0198 (4)
C3	0.33445 (14)	0.4627 (3)	0.36677 (12)	0.0202 (4)
C42	0.82336 (14)	1.0955 (3)	0.51858 (13)	0.0227 (4)

C62	0.33233 (14)	0.9082 (3)	0.49978 (13)	0.0241 (4)
C6	0.51354 (13)	0.3398 (3)	0.55560 (12)	0.0189 (4)
C29	−0.04957 (13)	0.5192 (3)	0.53528 (12)	0.0181 (4)
C24	−0.02878 (13)	0.5128 (3)	0.61509 (12)	0.0182 (4)
C8	0.57394 (13)	0.3606 (3)	0.45261 (12)	0.0184 (4)
C69	0.40732 (13)	0.9150 (3)	0.42277 (12)	0.0185 (4)
C7	0.58546 (13)	0.3319 (3)	0.53187 (12)	0.0183 (4)
C49	0.95774 (13)	1.0134 (3)	0.58130 (13)	0.0190 (4)
C48	1.04131 (14)	0.9686 (3)	0.63709 (13)	0.0217 (4)
C46	1.08429 (14)	0.9386 (3)	0.51791 (13)	0.0219 (4)
C30	−0.19092 (13)	0.5917 (3)	0.42252 (12)	0.0208 (4)
H30A	−0.195314	0.465826	0.394977	0.031*
H30B	−0.165253	0.690518	0.398138	0.031*
H30C	−0.248743	0.634908	0.418844	0.031*
C44	0.94005 (13)	1.0249 (3)	0.50165 (12)	0.0197 (4)
C66	0.58361 (14)	0.8529 (3)	0.46485 (13)	0.0206 (4)
C71	0.60075 (14)	0.8227 (3)	0.60429 (13)	0.0258 (4)
H71A	0.578830	0.903248	0.637924	0.039*
H71B	0.660364	0.861399	0.612352	0.039*
H71C	0.599411	0.683589	0.618036	0.039*
C31	0.07288 (15)	0.4634 (4)	0.75120 (13)	0.0263 (4)
H31A	0.049780	0.341920	0.765162	0.039*
H31B	0.046261	0.577200	0.766781	0.039*
H31C	0.135692	0.467660	0.778760	0.039*
C51	0.98219 (16)	0.9950 (4)	0.38378 (14)	0.0280 (5)
H51A	1.028745	1.063988	0.372886	0.042*
H51B	0.927512	1.065112	0.358458	0.042*
H51C	0.977081	0.860691	0.362901	0.042*
C50	0.86261 (17)	1.0583 (4)	0.66409 (15)	0.0284 (5)
C33	0.16380 (14)	0.4060 (4)	0.52176 (14)	0.0259 (5)
H33A	0.148733	0.294353	0.485177	0.039*
H33B	0.218050	0.377880	0.565271	0.039*
H33C	0.170850	0.524415	0.493788	0.039*
C53	1.19054 (14)	0.8887 (4)	0.64940 (15)	0.0301 (5)
H53A	1.225454	1.008608	0.656930	0.045*
H53B	1.214230	0.787698	0.624216	0.045*
H53C	1.191737	0.840579	0.700719	0.045*
C73	0.57111 (16)	0.8896 (4)	0.32820 (15)	0.0292 (5)
H73A	0.573261	0.754593	0.309962	0.044*
H73B	0.630055	0.941262	0.351905	0.044*
H73C	0.538002	0.972313	0.283348	0.044*
H5	0.3845 (19)	0.391 (4)	0.5203 (16)	0.022 (6)*
H9	0.4873 (17)	0.424 (4)	0.3426 (16)	0.017 (6)*
H22	−0.220 (2)	0.629 (4)	0.5596 (17)	0.030 (7)*
H1	0.282 (2)	0.521 (5)	0.2629 (19)	0.035 (8)*
H12	0.631 (2)	0.369 (4)	0.3832 (19)	0.031 (7)*
H62	0.284 (2)	0.912 (5)	0.5170 (18)	0.035 (8)*
H10	0.482 (3)	0.308 (6)	0.641 (2)	0.061 (11)*

C70	0.24418 (15)	0.9659 (4)	0.35795 (14)	0.0265 (4)
H70A	0.194877	0.947555	0.375199	0.040*
H70B	0.240186	0.871975	0.315813	0.040*
H70C	0.243510	1.100411	0.338199	0.040*
H42	0.767 (2)	1.131 (4)	0.5092 (18)	0.033 (7)*
H11	0.669 (2)	0.304 (5)	0.630 (2)	0.041 (9)*
H10G	0.392 (2)	0.449 (5)	0.6864 (19)	0.036 (8)*
H10E	0.425 (3)	0.802 (6)	0.728 (3)	0.062 (11)*
H10F	0.436 (2)	0.781 (6)	0.654 (2)	0.053 (10)*
H10A	0.718 (2)	0.192 (6)	0.760 (2)	0.056 (10)*
H10I	0.781 (2)	0.915 (5)	0.850 (2)	0.039 (8)*
H10C	0.568 (3)	0.622 (6)	0.746 (2)	0.055 (11)*
H10D	0.634 (2)	0.518 (5)	0.7604 (19)	0.034 (8)*
H10B	0.763 (2)	0.367 (5)	0.7558 (19)	0.033 (8)*
H10K	0.905 (3)	0.515 (7)	0.816 (3)	0.076 (14)*
H10L	0.881 (3)	0.528 (6)	0.738 (3)	0.072 (13)*
H10H	0.389 (3)	0.243 (7)	0.709 (3)	0.077 (13)*
H10J	0.714 (3)	0.832 (6)	0.788 (2)	0.057 (11)*
H50A	0.868 (2)	0.928 (5)	0.683 (2)	0.044 (9)*
H50B	0.803 (3)	1.094 (6)	0.651 (2)	0.063 (11)*
H50C	0.902 (3)	1.156 (5)	0.701 (2)	0.055 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O12	0.0148 (7)	0.0379 (8)	0.0204 (7)	0.0037 (6)	0.0089 (6)	0.0030 (6)
O74	0.0245 (8)	0.0298 (7)	0.0219 (7)	−0.0003 (6)	0.0102 (6)	0.0024 (6)
O10	0.0172 (7)	0.0413 (9)	0.0174 (7)	0.0025 (6)	0.0073 (6)	0.0042 (6)
O72	0.0165 (7)	0.0332 (8)	0.0358 (9)	0.0021 (6)	0.0117 (6)	−0.0004 (7)
O102	0.0174 (8)	0.0367 (9)	0.0231 (7)	−0.0010 (7)	0.0065 (6)	−0.0007 (7)
O11	0.0138 (7)	0.0391 (9)	0.0191 (7)	0.0056 (6)	0.0055 (6)	0.0018 (6)
O32	0.0144 (7)	0.0329 (8)	0.0264 (8)	0.0030 (6)	0.0030 (6)	−0.0036 (6)
O34	0.0196 (7)	0.0384 (9)	0.0206 (7)	0.0012 (6)	0.0081 (6)	−0.0031 (6)
O54	0.0235 (8)	0.0373 (8)	0.0233 (8)	0.0024 (7)	0.0049 (7)	−0.0005 (7)
O1	0.0149 (7)	0.0485 (10)	0.0214 (7)	0.0049 (7)	0.0031 (6)	0.0077 (7)
O100	0.0244 (9)	0.0339 (9)	0.0214 (8)	0.0019 (7)	0.0081 (7)	−0.0022 (7)
O105	0.0314 (9)	0.0353 (9)	0.0236 (8)	0.0057 (7)	0.0145 (7)	0.0029 (7)
O52	0.0197 (8)	0.0368 (9)	0.0378 (9)	0.0017 (6)	0.0154 (7)	−0.0023 (7)
O2	0.0150 (7)	0.0467 (10)	0.0282 (8)	0.0038 (7)	0.0075 (7)	0.0045 (7)
O104	0.0268 (8)	0.0426 (10)	0.0290 (8)	0.0020 (7)	0.0159 (7)	0.0003 (8)
O101	0.0279 (9)	0.0394 (10)	0.0288 (9)	−0.0025 (8)	0.0085 (7)	0.0033 (7)
N27	0.0135 (8)	0.0242 (8)	0.0233 (9)	0.0001 (6)	0.0078 (7)	−0.0025 (6)
N41	0.0172 (8)	0.0218 (8)	0.0262 (9)	0.0010 (7)	0.0104 (7)	−0.0017 (7)
N23	0.0164 (8)	0.0250 (8)	0.0203 (8)	0.0000 (7)	0.0074 (7)	−0.0001 (7)
N67	0.0185 (9)	0.0222 (8)	0.0236 (8)	0.0005 (7)	0.0122 (7)	0.0006 (7)
N21	0.0128 (8)	0.0232 (8)	0.0200 (8)	0.0001 (6)	0.0058 (7)	−0.0005 (6)
N61	0.0144 (8)	0.0278 (9)	0.0245 (9)	−0.0001 (7)	0.0094 (7)	0.0000 (7)
N63	0.0190 (9)	0.0281 (9)	0.0224 (8)	−0.0001 (7)	0.0111 (7)	0.0008 (7)

O103	0.0296 (10)	0.0955 (18)	0.0216 (9)	−0.0251 (11)	0.0102 (8)	−0.0029 (10)
N45	0.0164 (8)	0.0253 (9)	0.0234 (9)	0.0010 (7)	0.0086 (7)	−0.0011 (7)
N47	0.0121 (8)	0.0251 (8)	0.0270 (9)	0.0005 (7)	0.0046 (7)	−0.0026 (7)
N43	0.0155 (9)	0.0225 (8)	0.0268 (9)	0.0010 (6)	0.0047 (7)	−0.0012 (7)
N25	0.0146 (8)	0.0250 (8)	0.0187 (8)	0.0013 (6)	0.0038 (7)	0.0010 (6)
N65	0.0147 (8)	0.0231 (8)	0.0210 (8)	0.0011 (6)	0.0073 (7)	0.0015 (7)
C9	0.0166 (9)	0.0211 (9)	0.0177 (9)	−0.0003 (7)	0.0073 (8)	0.0000 (7)
C28	0.0156 (9)	0.0194 (9)	0.0208 (9)	−0.0013 (7)	0.0066 (8)	−0.0014 (7)
C68	0.0194 (10)	0.0170 (9)	0.0242 (10)	−0.0007 (7)	0.0110 (8)	−0.0004 (7)
C64	0.0168 (9)	0.0189 (9)	0.0223 (10)	−0.0015 (7)	0.0090 (8)	−0.0010 (7)
C5	0.0139 (9)	0.0215 (9)	0.0217 (10)	−0.0007 (7)	0.0084 (8)	−0.0001 (7)
C22	0.0156 (10)	0.0245 (10)	0.0221 (10)	−0.0005 (8)	0.0078 (8)	−0.0002 (8)
C4	0.0151 (9)	0.0179 (9)	0.0211 (9)	−0.0003 (7)	0.0066 (8)	−0.0006 (7)
C26	0.0152 (9)	0.0204 (9)	0.0227 (10)	−0.0003 (7)	0.0053 (8)	−0.0022 (8)
C3	0.0161 (9)	0.0222 (9)	0.0214 (10)	0.0000 (7)	0.0056 (8)	0.0008 (8)
C42	0.0134 (10)	0.0236 (10)	0.0307 (11)	0.0011 (7)	0.0076 (8)	−0.0013 (8)
C62	0.0176 (10)	0.0329 (11)	0.0252 (10)	0.0001 (8)	0.0118 (8)	0.0008 (8)
C6	0.0187 (9)	0.0199 (9)	0.0190 (9)	−0.0013 (7)	0.0078 (8)	−0.0009 (7)
C29	0.0128 (9)	0.0200 (9)	0.0206 (9)	−0.0006 (7)	0.0050 (7)	0.0001 (7)
C24	0.0146 (9)	0.0180 (9)	0.0218 (10)	0.0000 (7)	0.0063 (8)	0.0003 (7)
C8	0.0158 (9)	0.0196 (9)	0.0222 (10)	−0.0003 (7)	0.0097 (8)	−0.0005 (7)
C69	0.0151 (9)	0.0193 (9)	0.0225 (10)	0.0004 (7)	0.0086 (8)	0.0003 (7)
C7	0.0145 (9)	0.0196 (9)	0.0193 (9)	0.0016 (7)	0.0042 (7)	−0.0001 (7)
C49	0.0132 (9)	0.0206 (9)	0.0238 (10)	0.0003 (7)	0.0072 (8)	−0.0015 (7)
C48	0.0178 (10)	0.0189 (9)	0.0282 (11)	0.0002 (8)	0.0078 (9)	−0.0021 (8)
C46	0.0167 (10)	0.0204 (9)	0.0291 (11)	−0.0007 (7)	0.0089 (8)	−0.0022 (8)
C30	0.0144 (9)	0.0281 (10)	0.0175 (9)	0.0007 (7)	0.0028 (8)	0.0003 (8)
C44	0.0159 (9)	0.0184 (9)	0.0253 (10)	−0.0011 (7)	0.0078 (8)	−0.0025 (8)
C66	0.0187 (9)	0.0181 (9)	0.0271 (10)	0.0000 (7)	0.0107 (8)	−0.0003 (8)
C71	0.0199 (10)	0.0316 (11)	0.0235 (10)	0.0017 (9)	0.0048 (8)	0.0005 (9)
C31	0.0205 (10)	0.0381 (12)	0.0182 (10)	0.0022 (9)	0.0044 (8)	0.0024 (9)
C51	0.0279 (12)	0.0360 (12)	0.0226 (10)	−0.0008 (9)	0.0119 (9)	−0.0024 (9)
C50	0.0280 (12)	0.0328 (12)	0.0312 (12)	0.0035 (10)	0.0190 (10)	−0.0003 (10)
C33	0.0148 (10)	0.0335 (11)	0.0327 (12)	0.0018 (8)	0.0125 (9)	−0.0035 (9)
C53	0.0146 (10)	0.0324 (11)	0.0376 (13)	0.0031 (8)	0.0023 (9)	−0.0039 (10)
C73	0.0266 (12)	0.0373 (12)	0.0315 (12)	0.0016 (9)	0.0198 (10)	0.0014 (10)
C70	0.0175 (10)	0.0350 (11)	0.0255 (10)	0.0019 (9)	0.0056 (8)	0.0018 (9)

Geometric parameters (Å, º)

O12—C8	1.366 (2)	N43—C44	1.360 (3)
O12—H12	0.86 (3)	N25—C26	1.370 (3)
O74—C68	1.234 (3)	N25—C24	1.376 (3)
O10—C6	1.359 (2)	N25—C31	1.465 (3)
O10—H10	0.86 (4)	N65—C64	1.365 (3)
O72—C66	1.221 (3)	N65—C66	1.372 (3)
O102—H10A	0.89 (4)	N65—C71	1.465 (3)
O102—H10B	0.85 (4)	C9—C4	1.396 (3)

O11—C7	1.356 (2)	C9—C8	1.383 (3)
O11—H11	0.83 (4)	C9—H9	1.00 (3)
O32—C26	1.229 (3)	C28—C29	1.426 (3)
O34—C28	1.224 (3)	C68—C69	1.415 (3)
O54—C48	1.221 (3)	C64—C69	1.378 (3)
O1—C3	1.333 (3)	C5—C4	1.388 (3)
O1—H1	0.85 (3)	C5—C6	1.386 (3)
O100—H10C	0.83 (4)	C5—H5	0.97 (3)
O100—H10D	0.82 (4)	C22—H22	0.97 (3)
O105—H10E	0.86 (5)	C4—C3	1.481 (3)
O105—H10F	0.88 (4)	C42—H42	0.92 (3)
O52—C46	1.219 (3)	C62—H62	0.96 (3)
O2—C3	1.213 (3)	C6—C7	1.403 (3)
O104—H10G	0.91 (3)	C29—C24	1.364 (3)
O104—H10H	0.93 (5)	C8—C7	1.395 (3)
O101—H10I	0.94 (4)	C49—C48	1.425 (3)
O101—H10J	0.88 (4)	C49—C44	1.370 (3)
N27—C28	1.406 (3)	C30—H30A	0.9800
N27—C26	1.396 (3)	C30—H30B	0.9800
N27—C33	1.481 (3)	C30—H30C	0.9800
N41—C42	1.339 (3)	C71—H71A	0.9800
N41—C49	1.387 (3)	C71—H71B	0.9800
N41—C50	1.458 (3)	C71—H71C	0.9800
N23—C22	1.339 (3)	C31—H31A	0.9800
N23—C24	1.358 (3)	C31—H31B	0.9800
N67—C68	1.403 (3)	C31—H31C	0.9800
N67—C66	1.399 (3)	C51—H51A	0.9800
N67—C73	1.467 (3)	C51—H51B	0.9800
N21—C22	1.342 (3)	C51—H51C	0.9800
N21—C29	1.385 (3)	C50—H50A	0.94 (3)
N21—C30	1.456 (3)	C50—H50B	0.96 (4)
N61—C62	1.336 (3)	C50—H50C	1.00 (4)
N61—C69	1.385 (3)	C33—H33A	0.9800
N61—C70	1.461 (3)	C33—H33B	0.9800
N63—C64	1.353 (3)	C33—H33C	0.9800
N63—C62	1.338 (3)	C53—H53A	0.9800
O103—H10K	0.78 (5)	C53—H53B	0.9800
O103—H10L	0.84 (5)	C53—H53C	0.9800
N45—C46	1.374 (3)	C73—H73A	0.9800
N45—C44	1.375 (3)	C73—H73B	0.9800
N45—C51	1.466 (3)	C73—H73C	0.9800
N47—C48	1.412 (3)	C70—H70A	0.9800
N47—C46	1.396 (3)	C70—H70B	0.9800
N47—C53	1.471 (3)	C70—H70C	0.9800
N43—C42	1.336 (3)		

C8—O12—H12	107 (2)	N23—C24—C29	112.27 (18)
C6—O10—H10	108 (3)	C29—C24—N25	122.19 (18)

H10A—O102—H10B	109 (3)	O12—C8—C9	123.47 (18)
C7—O11—H11	111 (2)	O12—C8—C7	116.40 (18)
C3—O1—H1	111 (2)	C9—C8—C7	120.14 (18)
H10C—O100—H10D	97 (3)	N61—C69—C68	132.3 (2)
H10E—O105—H10F	109 (4)	C64—C69—N61	104.82 (17)
H10G—O104—H10H	105 (3)	C64—C69—C68	122.86 (19)
H10I—O101—H10J	99 (3)	O11—C7—C6	121.96 (18)
C28—N27—C33	116.29 (17)	O11—C7—C8	118.61 (18)
C26—N27—C28	126.45 (17)	C8—C7—C6	119.43 (18)
C26—N27—C33	117.24 (17)	N41—C49—C48	131.5 (2)
C42—N41—C49	106.10 (18)	C44—C49—N41	105.08 (18)
C42—N41—C50	126.3 (2)	C44—C49—C48	123.34 (19)
C49—N41—C50	127.5 (2)	O54—C48—N47	121.84 (19)
C22—N23—C24	103.11 (17)	O54—C48—C49	127.2 (2)
C68—N67—C73	117.64 (18)	N47—C48—C49	110.97 (19)
C66—N67—C68	126.75 (17)	O52—C46—N45	121.3 (2)
C66—N67—C73	115.61 (18)	O52—C46—N47	121.2 (2)
C22—N21—C29	106.10 (17)	N45—C46—N47	117.47 (18)
C22—N21—C30	126.55 (18)	N21—C30—H30A	109.5
C29—N21—C30	127.34 (17)	N21—C30—H30B	109.5
C62—N61—C69	106.06 (18)	N21—C30—H30C	109.5
C62—N61—C70	126.25 (19)	H30A—C30—H30B	109.5
C69—N61—C70	127.66 (18)	H30A—C30—H30C	109.5
C62—N63—C64	103.07 (18)	H30B—C30—H30C	109.5
H10K—O103—H10L	110 (5)	N43—C44—N45	126.21 (19)
C46—N45—C44	119.25 (18)	N43—C44—C49	111.87 (18)
C46—N45—C51	119.00 (18)	C49—C44—N45	121.92 (18)
C44—N45—C51	121.74 (18)	O72—C66—N67	121.06 (19)
C48—N47—C53	117.03 (19)	O72—C66—N65	121.9 (2)
C46—N47—C48	127.02 (18)	N65—C66—N67	117.06 (18)
C46—N47—C53	115.92 (18)	N65—C71—H71A	109.5
C42—N43—C44	103.33 (18)	N65—C71—H71B	109.5
C26—N25—C24	119.03 (17)	N65—C71—H71C	109.5
C26—N25—C31	120.56 (18)	H71A—C71—H71B	109.5
C24—N25—C31	120.40 (17)	H71A—C71—H71C	109.5
C64—N65—C66	119.72 (17)	H71B—C71—H71C	109.5
C64—N65—C71	120.76 (17)	N25—C31—H31A	109.5
C66—N65—C71	119.51 (18)	N25—C31—H31B	109.5
C4—C9—H9	122.5 (15)	N25—C31—H31C	109.5
C8—C9—C4	119.88 (18)	H31A—C31—H31B	109.5
C8—C9—H9	117.6 (15)	H31A—C31—H31C	109.5
O34—C28—N27	120.75 (18)	H31B—C31—H31C	109.5
O34—C28—C29	127.67 (19)	N45—C51—H51A	109.5
N27—C28—C29	111.58 (17)	N45—C51—H51B	109.5
O74—C68—N67	121.51 (19)	N45—C51—H51C	109.5
O74—C68—C69	126.7 (2)	H51A—C51—H51B	109.5
N67—C68—C69	111.75 (18)	H51A—C51—H51C	109.5
N63—C64—N65	126.15 (19)	H51B—C51—H51C	109.5

N63—C64—C69	112.08 (18)	N41—C50—H50A	108 (2)
N65—C64—C69	121.77 (18)	N41—C50—H50B	107 (3)
C4—C5—H5	122.4 (16)	N41—C50—H50C	108 (2)
C6—C5—C4	119.37 (19)	H50A—C50—H50B	107 (3)
C6—C5—H5	118.2 (16)	H50A—C50—H50C	115 (3)
N23—C22—N21	113.42 (19)	H50B—C50—H50C	111 (3)
N23—C22—H22	126.9 (17)	N27—C33—H33A	109.5
N21—C22—H22	119.4 (17)	N27—C33—H33B	109.5
C9—C4—C3	121.08 (18)	N27—C33—H33C	109.5
C5—C4—C9	120.66 (18)	H33A—C33—H33B	109.5
C5—C4—C3	118.26 (18)	H33A—C33—H33C	109.5
O32—C26—N27	120.64 (19)	H33B—C33—H33C	109.5
O32—C26—N25	121.57 (19)	N47—C53—H53A	109.5
N25—C26—N27	117.80 (18)	N47—C53—H53B	109.5
O1—C3—C4	112.93 (18)	N47—C53—H53C	109.5
O2—C3—O1	122.85 (19)	H53A—C53—H53B	109.5
O2—C3—C4	124.22 (19)	H53A—C53—H53C	109.5
N41—C42—H42	123 (2)	H53B—C53—H53C	109.5
N43—C42—N41	113.62 (19)	N67—C73—H73A	109.5
N43—C42—H42	123 (2)	N67—C73—H73B	109.5
N61—C62—N63	113.97 (19)	N67—C73—H73C	109.5
N61—C62—H62	122.8 (19)	H73A—C73—H73B	109.5
N63—C62—H62	123.2 (19)	H73A—C73—H73C	109.5
O10—C6—C5	123.93 (18)	H73B—C73—H73C	109.5
O10—C6—C7	115.58 (18)	N61—C70—H70A	109.5
C5—C6—C7	120.49 (19)	N61—C70—H70B	109.5
N21—C29—C28	131.99 (19)	N61—C70—H70C	109.5
C24—C29—N21	105.09 (17)	H70A—C70—H70B	109.5
C24—C29—C28	122.92 (18)	H70A—C70—H70C	109.5
N23—C24—N25	125.54 (18)	H70B—C70—H70C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O32^{i}	0.85 (3)	1.86 (3)	2.672 (2)	161 (3)
O10—H10···O104	0.86 (4)	1.79 (4)	2.643 (2)	174 (4)
O11—H11···O10	0.83 (3)	2.32 (4)	2.709 (2)	109 (3)
O11—H11···O102	0.83 (4)	1.88 (4)	2.680 (2)	161 (3)
O12—H12···O100^{i}	0.86 (3)	1.86 (3)	2.702 (2)	166 (3)
O100—H10*C*···O105	0.83 (4)	2.07 (4)	2.851 (3)	157 (4)
O100—H10*D*···O10	0.82 (4)	2.74 (3)	3.286 (2)	126 (3)
O100—H10*D*···O102	0.82 (4)	2.02 (4)	2.798 (3)	158 (3)
O101—H10*I*···N43^{ii}	0.94 (4)	1.97 (4)	2.898 (3)	170 (3)
O101—H10*J*···O100	0.88 (4)	2.09 (4)	2.960 (3)	170 (4)
O102—H10*A*···O101^{iii}	0.89 (4)	1.87 (4)	2.754 (3)	169 (4)
O102—H10*B*···O103	0.85 (4)	1.85 (4)	2.691 (3)	173 (3)
O103—H10*K*···O34^{iv}	0.78 (5)	2.06 (5)	2.832 (3)	168 (5)
O103—H10*L*···N23^{v}	0.84 (5)	1.99 (5)	2.823 (3)	168 (4)

O104—H10*G*···O1^{vi}	0.91 (3)	2.50 (3)	3.011 (2)	116 (2)
O104—H10*G*···O105	0.91 (3)	2.03 (3)	2.857 (3)	151 (3)
O104—H10*H*···O74^{vi}	0.93 (5)	2.00 (5)	2.924 (2)	171 (4)
O105—H10*E*···O74^{ii}	0.86 (5)	2.11 (5)	2.952 (2)	169 (4)
O105—H10*F*···N63	0.88 (4)	1.92 (4)	2.798 (2)	173 (4)
C22—H22···O72^{vii}	0.97 (3)	2.38 (3)	3.227 (3)	146 (2)
C30—H30*C*···O12^{vii}	0.98	2.71	3.247 (3)	115
C31—H31*C*···O2^{vi}	0.98	2.40	3.326 (3)	158
C42—H42···O12^{viii}	0.92 (3)	2.45 (3)	3.254 (3)	146 (3)
C42—H42···O72	0.92 (3)	2.66 (3)	3.136 (3)	113 (2)
C62—H62···O52^{vii}	0.96 (3)	2.24 (3)	3.119 (3)	151 (3)

Symmetry codes: (i) x, $-y+1$, $z-1/2$; (ii) x, $-y+2$, $z+1/2$; (iii) x, $y-1$, z; (iv) $x+1$, $-y+1$, $z+1/2$; (v) $x+1$, y, z; (vi) x, $-y+1$, $z+1/2$; (vii) $x-1$, y, z; (viii) x, $y+1$, z.