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Synthesis and structure of a 1:1 cocrystal of *N*,*N*′-bis(pyridin-3-ylmethyl)pyromellitic diimide and naphthalene-2,6-dicarboxylic acid

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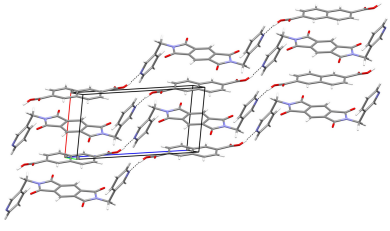
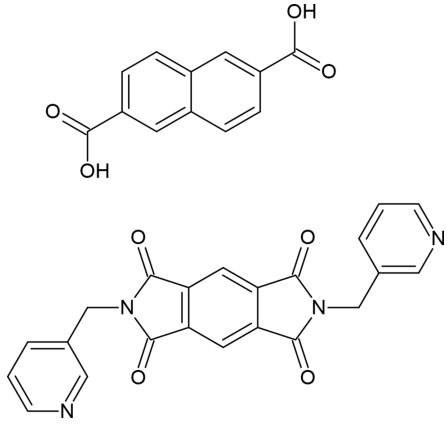
In the title cocrystal, $C_{22}H_{14}N_4O_4{\cdot}C_{12}H_8O_4$, both components are completed by crystallographic inversion symmetry and the dihedral angle between the central fused ring system and the pendant pyridine ring is 66.21 (5)°. In the extended structure, the components are linked by O—H···N hydrogen bonds, generating [001] chains, and the packing is consolidated by π–π stacking and C—H···O interactions.

1. Chemical context

N,*N*′-Bis(pyridin-3-ylmethyl)pyromellitic diimide, $C_{22}H_{14}N_4O_4$, (Lig1) consists of a pyromellitic diimide core linked to *meta*-substituted pyridyl groups *via* –CH_2– linkages. These rotatable linkages impart conformational flexibility, allowing Lig1 to adopt Z_C- and Z_T- modes, where *Z* denotes an *anti* orientation of the pyridyl rings, while *C* (*cis*) and *T* (*trans*) describe the relative positions of the pyridyl nitrogen atoms (Yan *et al.*, 2010). The pyromellitic diimide core further promotes π–π stacking interactions, contributing to supramolecular assembly. Owing to its semi-rigid nature, Lig1 has been widely employed in the construction of metal–organic frameworks (MOFs) exhibiting diverse topologies, with potential applications in gas sorption (Li *et al.*, 2012) and fluorescence (Huang *et al.*, 2020; Li *et al.*, 2018).

Deprotonated naphthalene dicarboxylic acid ($C_{12}H_8O_4$; NDC) ligands are also widely used to prepare MOFs with potential applications in gas storage, gas separation, luminescence and catalysis (Gangu *et al.*, 2017). Their aromatic ring system allows for increased π–π stacking interactions, enhancing supramolecular recognition and enabling the formation of complex polymer networks.





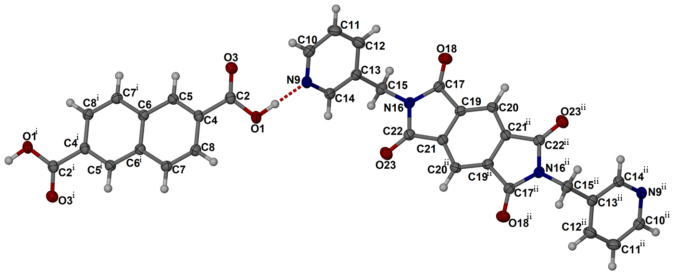


Figure 1
The molecular structure of (**I**) with displacement ellipsoids drawn at the 70% probability level. [Symmetry codes: (i) $2-x, 1-y, 2-z$; (ii) $1-x, 1-y, -z$].

The aim of this work was to prepare a zinc mixed-ligand MOF containing Lig1 and NDC. However, single-crystal X-ray diffraction (SCXRD) revealed that a 1:1 cocrystal of Lig1 and NDC, (**I**), had formed from the solvothermal reaction and we now describe its structure.

2. Structural commentary

Compound (**I**) crystallizes in the triclinic space group $P\overline{1}$ with half a molecule of Lig1 and half a molecule of NDC in the asymmetric unit (Fig. 1). Both complete molecules are generated by crystallographic inversion centres at (1/2, 1/2, 0) and (1, 1/2, 1) for the asymmetric atoms of Lig1 and NDC, respectively. The Lig1 molecule adopts a Z_T- mode and the dihedral angle between the central fused ring system and the pendant pyridine ring is 66.21 (5)°.

3. Supramolecular features

In the extended structure of (**I**), the NDC molecule links to Lig1 *via* an O1—H1···N9 hydrogen bond between the carboxylic acid group of NDC and the py-*N* of Lig1, and a secondary interaction between the Ar-H atom of Lig 1 and the C═O group of the NDC molecule (C10—H10···O3). These interactions result in extended chains of alternating Lig1 and NDC molecules running along the crystallographic *c*-axis direction (Table 1, Fig. 2).

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N9	0.95 (1)	1.64 (1)	2.5806 (13)	172 (2)
C15—H15A···O3^{i}	0.99	2.56	3.4073 (14)	143
C11—H11···O23^{ii}	0.95	2.51	3.2402 (15)	134
C10—H10···O3^{iii}	0.95	2.64	3.2414 (15)	122
C11—H11···O3^{iii}	0.95	2.62	3.2207 (15)	122

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

The chains stack in the *ac* plane in an offset arrangement facilitated by π–π stacking interactions [centroid-to-centroid distance = 3.767 (1) Å] between the pyridyl moieties of Lig1 of adjacent chains, as well as π–π interactions [centroid-to-centroid distance = 3.761 (1) Å] between the pyromellitic moiety of Lig1 and the naphthalene moiety of NDC of adjacent chains. Additionally, the chains interact with neighboring chains *via* C—H···O interactions in the *ac* plane (C15—H15A···O3) as well as in the *b*-axis direction (C11—H11···O23) (Fig. 2).

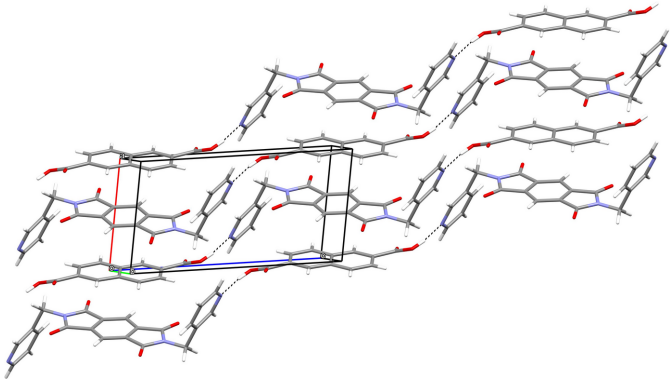


Figure 2
The packing of (**I**) viewed down the *b*-axis direction.

4. Hirshfeld surface analysis

To further quantify the nature and relative contributions of intermolecular interactions within the crystal structure of (**I**), two-dimensional fingerprint plots were generated using *CrystalExplorer* (Spackman *et al.* 2021). The Hirshfeld surface was constructed from a hydrogen-bonded unit comprising one NDC and one Lig1 molecule. The breakdown of intermolecular contacts and their percentage contributions to the total Hirshfeld surface are presented in Fig. 3. The contributions follow the order O···H/H···O (32%) > H···H (29%) > C···C (14%) > C···H/H···C (13%) > N···H/H···N (5%) > C···O/O···C (2.9%). Although O···H/H···O interactions make the largest contribution to the Hirshfeld surface, these contacts are relatively long, with the closest atom–atom distance of approximately 2.3 Å, and are mainly associated

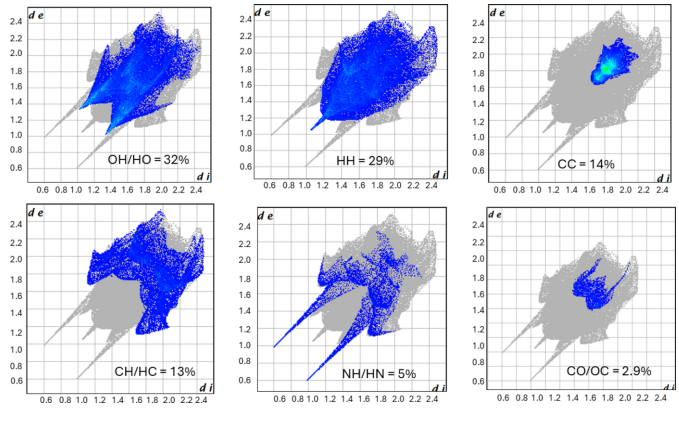


Figure 3
Two-dimensional fingerprint plots for (**I**) showing the various contributions to the Hirshfeld surface.

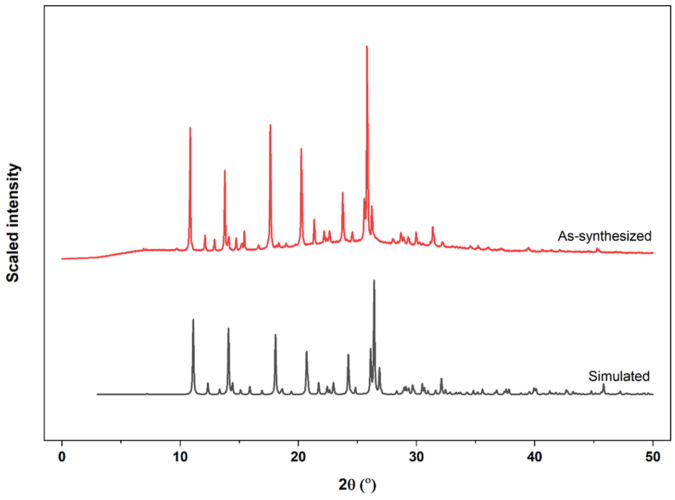


Figure 4
Experimental PXRD pattern of (**I**) (red) overlaid with the simulated pattern generated from SCXRD data (black), confirming phase purity.

with weaker C—H···O interactions. H···H contacts also contribute significantly, indicating that van der Waals interactions play an important role in consolidating the crystal structure. The C···C and C···H interactions contribute 14% and 13%, respectively, and can be attributed to π–π stacking and C-H···π interactions. N···H/H···N interactions, associated with O—H···N hydrogen bonding, contribute a smaller proportion (5%) but represent the shortest intermolecular contacts, with a closest atom–atom distance of approximately 1.6 Å.

5. Thermogravimetric analysis (TGA) and powder X-ray diffraction (PXRD)

PXRD analysis was performed to assess the bulk phase purity of (**I**). A comparison of the PXRD pattern of the as-synthesized sample with the simulated pattern of (**I**) shows good agreement, which indicates phase purity of the sample (Fig. 4). The TGA curve shows that the cocrystal decomposes at 290 °C (Fig. 5).

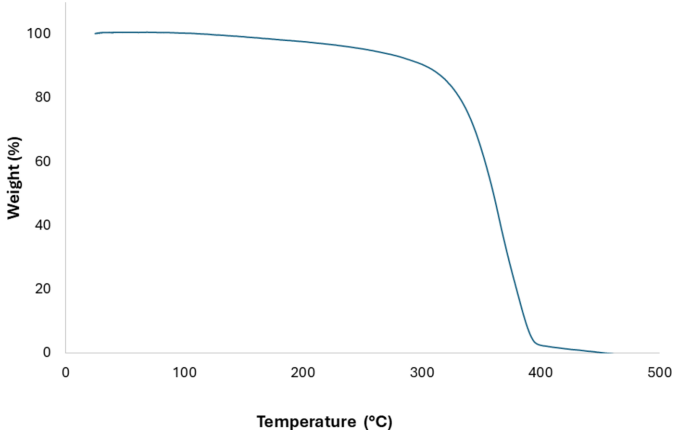


Figure 5
TGA curve of (**I**). Decomposition starts at 290 °C.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{14}N_4O_4{\cdot}C_{12}H_8O_4$
M_r	614.55
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	130
a, b, c (Å)	6.9314 (2), 8.3554 (3), 12.5463 (4)
α, β, γ (°)	79.716 (1), 80.977 (1), 74.432 (1)
V (Å^3)	684.15 (4)
Z	1
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.28 × 0.17 × 0.13
Data collection	
Diffractometer	BRUKER D8 QUEST
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.650, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23721, 3391, 3048
R_{int}	0.040
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.107, 1.10
No. of reflections	3391
No. of parameters	211
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.28

Computer programs: *APEX4* and *SAINT* (Bruker, 2022), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2019/3* (Sheldrick, 2015*b*) and *X-SEED* (Barbour, 2020).

6. Database survey

A search of the Cambridge Structural Database (CSD) (Groom *et al.*, 2016) showed that Lig1 has been used mostly in the preparation of coordination complexes. Of the 20 crystal structures deposited Co [DIBBAU, DIBBEY, DIBBIC (Li *et al.*, 2018), OWEYEV (Li *et al.*, 2011)], Zn [OWEYUL (Li *et al.*, 2011), FISCEQ (Lü *et al.* 2005*b*), PALYIL (Lü *et al.* 2005*a*)], Cd [FISCAM (Lü *et al.* 2005*b*), PADHOT (Chai *et al.* 2010), PALYUX (Lü *et al.* 2005*a*), ZAVQIZ (Li *et al.*, 2017), ZAXSOI (Li *et al.*, 2012)], Ag (QAHBEI, QAHBIM; Yan *et al.*, 2011), Hg [HUHZUI (Huang *et al.*, 2020), PEVYUL (Li *et al.*, 2007)], Ni (HUJBAS; Huang *et al.*, 2020) and Mn [OWEXOE (Li *et al.*, 2011), ZAVQEV Li *et al.*, 2017)] and one entry is a salt of protonated Lig1 and a perchlorate ion (FISBUF; Lü *et al.*, 2005). The coordination complexes exhibit structural diversity, with some investigated for CO_2 sorption (Li *et al.*, 2012) and fluorescence properties (Huang *et al.*, 2020; Li *et al.*, 2018). A search on the CSD for NDC produced 683 coordination complexes containing NDC either by itself or in combination with other ligands. Additionally, there are 40 hits where NDC features in cocrystals or salts.

7. Synthesis and crystallization

Lig1 was synthesized according to a reported procedure (Li *et al.* 2009). Compound (**I**) crystallized from a solvothermal reaction of Lig1 (10 mg, 0.025 mmol), NDC (16 mg,

0.074 mmol), and $Zn(NO_3)_2·6H_2O$ (20 mg, 0.067 mmol) in 3 ml of *N*,*N*-dimethylformamide (DMF) at 373 K. Cream crystals of (**I**) formed after 3 days. The vial was then removed from the oven and washed with 2 ml of DMF.

8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bound H atoms were positioned (C—H = 0.95–0.99 Å) geometrically and refined as riding U_{iso}(H) = 1.2U_{eq}(C). The OH H atom was found in a difference map and refined with U_{iso}(H) = 1.5U_{eq}(O).

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Synthesis and structure of a 1:1 cocrystal of *N*,*N*′-bis(pyridin-3-ylmethyl)-pyromellitic diimide and naphthalene-2,6-dicarboxylic acid

Luccile Mbonzhe and Eustina Batisai

Computing details

N,N′-Bis(pyridin-3-ylmethyl)pyromellitic diimide–naphthalene-2,6-dicarboxylic acid (1/1)

Crystal data

$C_{22}H_{14}N_4O_4·C_{12}H_8O_4$
M_r = 614.55
Triclinic, $P\overline{1}$
a = 6.9314 (2) Å
b = 8.3554 (3) Å
c = 12.5463 (4) Å
α = 79.716 (1)°
β = 80.977 (1)°
γ = 74.432 (1)°
V = 684.15 (4) Å^3
Z = 1
$F(000)$ = 318
D_x = 1.492 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9965 reflections
θ = 2.6–28.3°
μ = 0.11 mm^{-1}
T = 130 K
Block, yellow
0.28 × 0.17 × 0.13 mm

Data collection

BRUKER D8 QUEST diffractometer
Radiation source: sealed tube
Detector resolution: 7.39 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.650, T_{max} = 0.746
23721 measured reflections
3391 independent reflections
3048 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 28.3°, θ_{min} = 2.6°
h = −9→8
k = −11→11
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.107
S = 1.10
3391 reflections
211 parameters
1 restraint
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 0.3031P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.94933 (14)	0.56834 (11)	0.61477 (7)	0.0239 (2)
H1	0.887 (2)	0.6401 (17)	0.5555 (9)	0.036*
O18	0.26446 (15)	0.92395 (11)	0.09152 (7)	0.0272 (2)
O23	0.50906 (14)	0.42909 (11)	0.30621 (7)	0.0243 (2)
O3	0.94674 (14)	0.81605 (11)	0.66050 (7)	0.0233 (2)
N9	0.74937 (16)	0.76234 (12)	0.46376 (8)	0.0198 (2)
N16	0.36780 (14)	0.69532 (12)	0.22228 (8)	0.0174 (2)
C7	1.08872 (17)	0.31773 (14)	0.92125 (9)	0.0174 (2)
H7	1.139624	0.199353	0.934264	0.021*
C8	1.06660 (16)	0.39725 (14)	0.81661 (9)	0.0171 (2)
H8	1.098256	0.333481	0.757836	0.021*
C4	0.99645 (16)	0.57470 (14)	0.79612 (9)	0.0161 (2)
C2	0.96390 (16)	0.66531 (14)	0.68329 (9)	0.0175 (2)
C14	0.59674 (18)	0.72163 (14)	0.43038 (9)	0.0199 (2)
H14	0.570054	0.615645	0.459367	0.024*
C13	0.47578 (17)	0.82771 (14)	0.35519 (9)	0.0170 (2)
C15	0.30468 (17)	0.77764 (14)	0.31994 (9)	0.0188 (2)
H15B	0.252472	0.700467	0.379958	0.023*
H15A	0.193824	0.878866	0.305142	0.023*
C17	0.33909 (17)	0.77633 (14)	0.11648 (9)	0.0182 (2)
C19	0.41928 (16)	0.64345 (13)	0.04451 (9)	0.0161 (2)
C20	0.42361 (17)	0.65689 (14)	−0.06757 (9)	0.0173 (2)
H20A	0.373241	0.760065	−0.111895	0.021*
C21	0.49260 (16)	0.49242 (13)	0.10978 (9)	0.0161 (2)
C10	0.78669 (18)	0.91129 (15)	0.42350 (10)	0.0203 (2)
H10	0.893561	0.940894	0.448094	0.024*
C11	0.67585 (19)	1.02447 (15)	0.34729 (11)	0.0239 (3)
H11	0.707520	1.128836	0.318970	0.029*
C12	0.51751 (18)	0.98197 (15)	0.31325 (10)	0.0221 (2)
H12	0.438083	1.057794	0.261594	0.027*
C22	0.46261 (16)	0.52553 (14)	0.22495 (9)	0.0171 (2)
C5	0.94801 (16)	0.66816 (14)	0.88063 (9)	0.0168 (2)
H5	0.903446	0.786937	0.865789	0.020*
C6	0.96400 (16)	0.58878 (13)	0.98974 (9)	0.0157 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0342 (5)	0.0199 (4)	0.0182 (4)	−0.0031 (3)	−0.0112 (3)	−0.0029 (3)

O18	0.0375 (5)	0.0159 (4)	0.0253 (4)	0.0007 (4)	−0.0065 (4)	−0.0037 (3)
O23	0.0314 (5)	0.0212 (4)	0.0178 (4)	−0.0035 (3)	−0.0039 (3)	−0.0003 (3)
O3	0.0315 (5)	0.0200 (4)	0.0205 (4)	−0.0099 (3)	−0.0075 (3)	0.0012 (3)
N9	0.0262 (5)	0.0188 (5)	0.0152 (4)	−0.0048 (4)	−0.0051 (4)	−0.0029 (3)
N16	0.0195 (4)	0.0170 (4)	0.0165 (4)	−0.0040 (3)	−0.0025 (3)	−0.0044 (3)
C7	0.0182 (5)	0.0158 (5)	0.0183 (5)	−0.0031 (4)	−0.0025 (4)	−0.0036 (4)
C8	0.0175 (5)	0.0184 (5)	0.0160 (5)	−0.0041 (4)	−0.0015 (4)	−0.0045 (4)
C4	0.0147 (5)	0.0187 (5)	0.0152 (5)	−0.0047 (4)	−0.0029 (4)	−0.0015 (4)
C2	0.0156 (5)	0.0204 (5)	0.0168 (5)	−0.0048 (4)	−0.0027 (4)	−0.0021 (4)
C14	0.0280 (6)	0.0173 (5)	0.0154 (5)	−0.0077 (4)	−0.0035 (4)	−0.0013 (4)
C13	0.0189 (5)	0.0170 (5)	0.0149 (5)	−0.0033 (4)	−0.0004 (4)	−0.0050 (4)
C15	0.0188 (5)	0.0206 (5)	0.0179 (5)	−0.0047 (4)	−0.0003 (4)	−0.0067 (4)
C17	0.0194 (5)	0.0171 (5)	0.0187 (5)	−0.0043 (4)	−0.0030 (4)	−0.0039 (4)
C19	0.0160 (5)	0.0143 (5)	0.0184 (5)	−0.0041 (4)	−0.0024 (4)	−0.0026 (4)
C20	0.0188 (5)	0.0145 (5)	0.0182 (5)	−0.0038 (4)	−0.0035 (4)	−0.0008 (4)
C21	0.0162 (5)	0.0162 (5)	0.0166 (5)	−0.0053 (4)	−0.0026 (4)	−0.0015 (4)
C10	0.0217 (5)	0.0205 (5)	0.0203 (5)	−0.0062 (4)	−0.0028 (4)	−0.0051 (4)
C11	0.0266 (6)	0.0168 (5)	0.0289 (6)	−0.0074 (4)	−0.0060 (5)	0.0008 (4)
C12	0.0229 (6)	0.0177 (5)	0.0243 (6)	−0.0032 (4)	−0.0059 (4)	0.0007 (4)
C22	0.0169 (5)	0.0167 (5)	0.0183 (5)	−0.0049 (4)	−0.0020 (4)	−0.0032 (4)
C5	0.0173 (5)	0.0161 (5)	0.0171 (5)	−0.0041 (4)	−0.0033 (4)	−0.0014 (4)
C6	0.0141 (5)	0.0167 (5)	0.0167 (5)	−0.0036 (4)	−0.0023 (4)	−0.0030 (4)

Geometric parameters (Å, º)

O1—C2	1.3135 (14)	C13—C12	1.3900 (16)
O1—H1	0.9501 (10)	C13—C15	1.5080 (15)
O18—C17	1.2073 (14)	C15—H15B	0.9900
O23—C22	1.2094 (14)	C15—H15A	0.9900
O3—C2	1.2182 (14)	C17—C19	1.4921 (15)
N9—C10	1.3335 (15)	C19—C20	1.3867 (15)
N9—C14	1.3391 (15)	C19—C21	1.3924 (15)
N16—C22	1.3911 (14)	C20—C21^{ii}	1.3872 (15)
N16—C17	1.3963 (14)	C20—H20A	0.9500
N16—C15	1.4619 (14)	C21—C22	1.4917 (15)
C7—C8	1.3732 (15)	C10—C11	1.3843 (17)
C7—C6^{i}	1.4216 (15)	C10—H10	0.9500
C7—H7	0.9500	C11—C12	1.3870 (17)
C8—C4	1.4201 (15)	C11—H11	0.9500
C8—H8	0.9500	C12—H12	0.9500
C4—C5	1.3748 (15)	C5—C6	1.4179 (15)
C4—C2	1.5013 (15)	C5—H5	0.9500
C14—C13	1.3889 (16)	C6—C6^{i}	1.422 (2)
C14—H14	0.9500		
C2—O1—H1	106.8 (11)	O18—C17—C19	128.73 (11)
C10—N9—C14	118.72 (10)	N16—C17—C19	105.69 (9)
C22—N16—C17	112.33 (9)	C20—C19—C21	122.87 (10)

C22—N16—C15	123.31 (9)	C20—C19—C17	129.04 (10)
C17—N16—C15	124.37 (9)	C21—C19—C17	108.09 (10)
C8—C7—C6^{i}	120.49 (10)	C19—C20—C21^{ii}	114.62 (10)
C8—C7—H7	119.8	C19—C20—H20A	122.7
C6^{i}—C7—H7	119.8	C21^{ii}—C20—H20A	122.7
C7—C8—C4	120.09 (10)	C20^{ii}—C21—C19	122.52 (10)
C7—C8—H8	120.0	C20^{ii}—C21—C22	129.54 (10)
C4—C8—H8	120.0	C19—C21—C22	107.95 (9)
C5—C4—C8	120.49 (10)	N9—C10—C11	122.60 (11)
C5—C4—C2	117.92 (10)	N9—C10—H10	118.7
C8—C4—C2	121.54 (10)	C11—C10—H10	118.7
O3—C2—O1	124.33 (10)	C10—C11—C12	118.46 (11)
O3—C2—C4	121.82 (10)	C10—C11—H11	120.8
O1—C2—C4	113.81 (10)	C12—C11—H11	120.8
N9—C14—C13	122.82 (11)	C11—C12—C13	119.57 (11)
N9—C14—H14	118.6	C11—C12—H12	120.2
C13—C14—H14	118.6	C13—C12—H12	120.2
C14—C13—C12	117.83 (11)	O23—C22—N16	125.12 (10)
C14—C13—C15	121.26 (10)	O23—C22—C21	128.96 (10)
C12—C13—C15	120.91 (10)	N16—C22—C21	105.93 (9)
N16—C15—C13	111.95 (9)	C4—C5—C6	120.56 (10)
N16—C15—H15B	109.2	C4—C5—H5	119.7
C13—C15—H15B	109.2	C6—C5—H5	119.7
N16—C15—H15A	109.2	C5—C6—C7^{i}	121.66 (10)
C13—C15—H15A	109.2	C5—C6—C6^{i}	118.99 (12)
H15B—C15—H15A	107.9	C7^{i}—C6—C6^{i}	119.35 (12)
O18—C17—N16	125.59 (11)		

C6^{i}—C7—C8—C4	1.93 (17)	C17—C19—C20—C21^{ii}	−179.30 (11)
C7—C8—C4—C5	−0.73 (17)	C20—C19—C21—C20^{ii}	0.13 (19)
C7—C8—C4—C2	−177.93 (10)	C17—C19—C21—C20^{ii}	179.46 (10)
C5—C4—C2—O3	18.94 (16)	C20—C19—C21—C22	−179.81 (10)
C8—C4—C2—O3	−163.79 (11)	C17—C19—C21—C22	−0.48 (12)
C5—C4—C2—O1	−158.88 (10)	C14—N9—C10—C11	−1.02 (18)
C8—C4—C2—O1	18.39 (15)	N9—C10—C11—C12	1.23 (19)
C10—N9—C14—C13	0.25 (17)	C10—C11—C12—C13	−0.65 (18)
N9—C14—C13—C12	0.28 (17)	C14—C13—C12—C11	−0.06 (17)
N9—C14—C13—C15	−179.75 (10)	C15—C13—C12—C11	179.98 (11)
C22—N16—C15—C13	83.09 (13)	C17—N16—C22—O23	177.96 (11)
C17—N16—C15—C13	−97.45 (12)	C15—N16—C22—O23	−2.52 (18)
C14—C13—C15—N16	−92.23 (12)	C17—N16—C22—C21	−1.83 (12)
C12—C13—C15—N16	87.73 (13)	C15—N16—C22—C21	177.69 (9)
C22—N16—C17—O18	−178.33 (12)	C20^{ii}—C21—C22—O23	1.7 (2)
C15—N16—C17—O18	2.15 (19)	C19—C21—C22—O23	−178.40 (12)
C22—N16—C17—C19	1.54 (12)	C20^{ii}—C21—C22—N16	−178.55 (11)
C15—N16—C17—C19	−177.97 (10)	C19—C21—C22—N16	1.39 (12)
O18—C17—C19—C20	−1.4 (2)	C8—C4—C5—C6	−1.07 (16)
N16—C17—C19—C20	178.69 (11)	C2—C4—C5—C6	176.22 (10)

O18—C17—C19—C21	179.28 (12)	C4—C5—C6—C7^{i}	−178.81 (10)
N16—C17—C19—C21	−0.59 (12)	C4—C5—C6—C6^{i}	1.64 (19)
C21—C19—C20—C21^{ii}	−0.12 (18)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N9	0.95 (1)	1.64 (1)	2.5806 (13)	172 (2)
C15—H15*A*···O3^{iii}	0.99	2.56	3.4073 (14)	143
C11—H11···O23^{iv}	0.95	2.51	3.2402 (15)	134
C10—H10···O3^{v}	0.95	2.64	3.2414 (15)	122
C11—H11···O3^{v}	0.95	2.62	3.2207 (15)	122

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