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# 2,2′-{(1*E*,1′*E*)-[Ethane-1,2-diylbis(azanylylidene)]-bis(methanylylidene)}bis(4-iodophenol)

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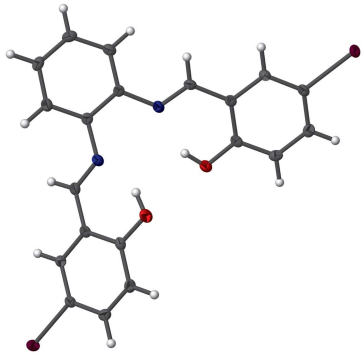
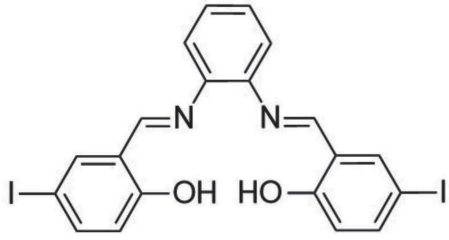
**Structural data**: full structural data are available from iucrdata.iucr.org

The title compound, $C_{20}H_{14}I_2N_2O_2$, a diiodo-Schiff base, crystallizes in space group *Pbca* with one molecule per asymmetric unit. The molecular structure reveals two intramolecular O—H···N hydrogen bonds that give the molecule a twisted structure with non-coplanar rings. In the crystal structure, the molecular packing is stabilized by $\pi$–$\pi$ stacking, hydrogen- and halogen-bonding (C—H···I; O···I) interactions.

**3D view**



**Chemical scheme**




## Structure description

2,2′-{(1*E*,1′*E*)-[Ethane-1,2-diylbis(azanylylidene)]bis(methanylylidene)}bis(4-iodophenol) (I) ($C_{20}H_{14}I_2N_2O_2$, Fig. 1) is a salen-type ligand under investigation in our laboratory for possible antimicrobial effects (Ceramella *et al.*, 2022) and the ability to bind to lanthanide metals, which could have other important medicinal properties (Kaczmarek *et al.*, 2018). As a result of the wide range of medical applications for such compounds, and in a continuation of our work in this area (Reimann *et al.*, 2019), the title compound (I) was prepared and its crystal structure is reported here.

Each molecule of (I) consists of a central ring (C8–C13) with *ortho* imine nitrogen atoms (N1 and N2) covalently bound through the imine C atoms, C7 and C14 respectively, to I1—Ar(O1—H1) and I2—Ar(O2—H2) rings (Fig. 1). Two intramolecular hydrogen bonds (Table 1), O1—H1···N1 [1.842 (15) Å, 152 (3)°] and O2—H2···N2 [1.806 (15) Å, 153 (3)°], result in an overall non-planar molecule with the I—Ar(OH) ring planes twisted with respect to the central ring (C8–13) plane [24.97 (7)° *versus* I1—Ar(O1—H1), and 39.37 (5)° *versus* I2—Ar(O2—H2)] (Fig. 1). The intramolecular hydrogen bonds of (I) show similarity to those of the dichlorinated Schiff base 3,5-dichloro-*N*-[2-(methylthio)phenyl]salicylaldimine (Hamaker *et al.*, 2010). Halogen bonding (I1···O2) and slipped $\pi$–$\pi$ stacking interactions stabilize the packing pattern. Along the *a*-axis direction, adjacent molecules related by $2_1$ screw-axis symmetry form

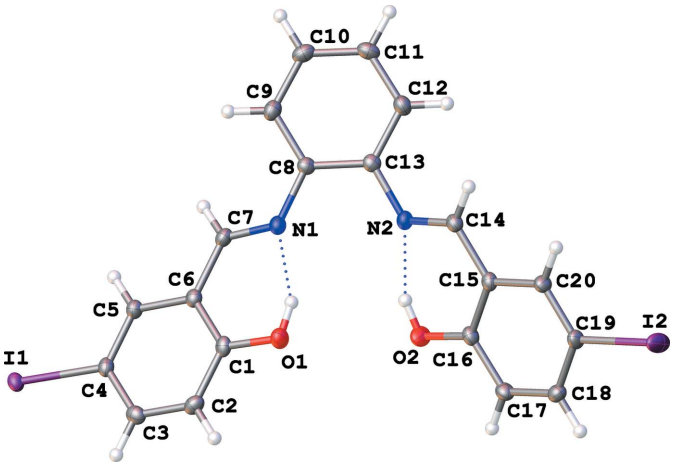



**Figure 1**
Molecular structure of (I) depicting the two intramolecular hydrogen bonds (O1—H1···N1 and O2—H2···N2). Displacement ellipsoids are shown at the 50% probability level

2-stacks/molecule of non-coplanar and alternating central ring (C8–C13) to I2—Ar(O2—H2) ring interactions (Fig. 2). This results in alternating longer [3.821 (13) Å] and shorter [3.734 (13) Å] central ring (C8–C13) centroid to I2—Ar(O2—H2) ring centroid distances with the corresponding alternating centroids slipped by 1.583 (4) and 1.548 (3) Å with respect to each other (Fig. 2). The additional hydrogen bonding provides three shorter and one longer C—H···I type interactions in which I2 has a larger displacement ellipsoid than I1 [C18—H18···I1 = 3.16 Å (159°), C5—H5···I2 = 3.17 Å (156°), C7—H7···I2 = 3.23 Å (152°), and C2—H2···I2 3.32 Å (131°)]. The two C—I bond lengths of (I) are similar to the C—I bond of 2,3,5,6-tetrafluoro-1,4-diiodobenzene (Tan & Tiekink, 2019). Along the *b*-axis direction, adjacent molecules appear to be arranged in a H (head, central C8–C13 ring) to T [tail, I1—Ar(O1—H1) and I2—Ar(O2—H2) rings] repeating pattern with the I1 and I2 atoms of the I1—Ar(O1—H1) and I2—Ar(O2—H2) rings interdigitated along the *c*-axis direction (Fig. 3).

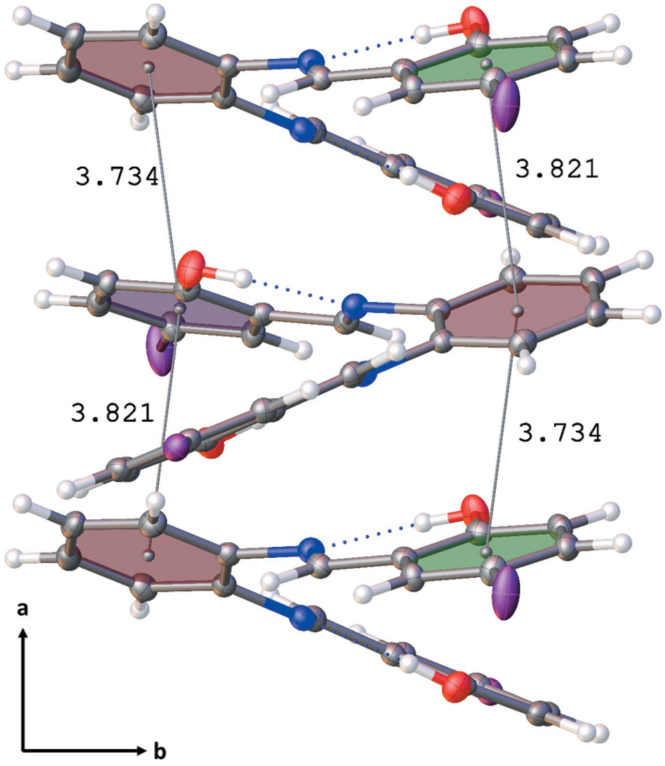



**Figure 2**
Diagram of the basic packing motif along the *a*-axis direction depicting the slipped ring $\pi$–$\pi$ interactions.

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2*A*···I2$^{i}$ | 0.95 | 3.32 | 4.009 (2) | 131 |
| C5—H5···I2$^{ii}$ | 0.95 | 3.17 | 4.061 (2) | 156 |
| C7—H7···I2$^{ii}$ | 0.95 | 3.23 | 4.094 (2) | 152 |
| C18—H18···I1$^{iii}$ | 0.95 | 3.16 | 4.066 (2) | 159 |
| O1—H1···N1 | 0.84 (1) | 1.84 (2) | 2.609 (3) | 152 (3) |
| O2—H2···N2 | 0.84 (1) | 1.81 (2) | 2.580 (3) | 153 (3) |

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

## Synthesis and crystallization

To a solution of 2-hydroxy-5-iodobenzaldehyde (4.60 g, 18.5 mmol) in ethanol (200 ml) was added an ethanol (10 ml) solution of *o*-phenylenediamine (1.00 g, 9.20 mmol), and the reaction mixture brought to reflux with vigorous stirring for 2 h. Upon cooling, the title compound (I) precipitated as an orange solid, and was filtered, washed with ethanol and dried under vacuum. Crystals of (I) suitable for single-crystal X-ray diffraction were grown from acetone layered with hexane. Yield: 4.37 g (83%), m.p. 212–214°C. $^{1}$H NMR (500 MHz, $CDCl_3$) $\delta$ 8.54 (*s*, 2H), 7.73–7.54 (*m*, 4H), 7.37 (*q*, *J* = 3.2 Hz, 2H), 7.22 (*q*, *J* = 3.2 Hz, 2H), 6.84 (*d*, *J* = 9.0 Hz, 2H). [Note: phenolic H atoms (2Hs) undergo rapid exchange thus their NMR signals are broadened into the baseline beyond recognition as reported (Charisiadis *et al.*, 2014).] $^{13}$C NMR

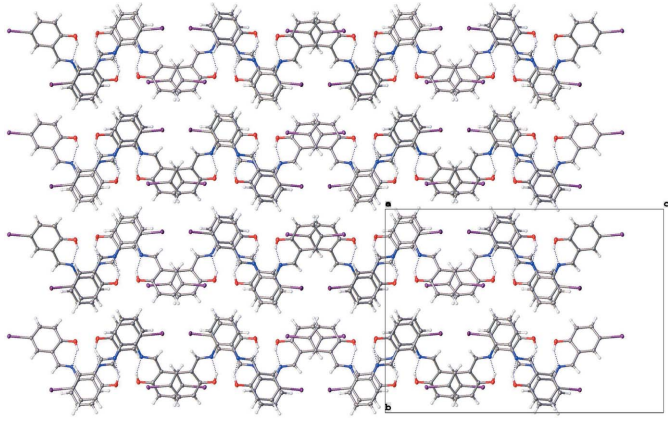


**Figure 3**
Molecular packing view of (I) along the *a*-axis direction. Displacement ellipsoids are shown at the 50% probability level.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{20}H_{14}I_2N_2O_2$ |
| $M_r$ | 568.13 |
| Crystal system, space group | Orthorhombic, *Pbca* |
| Temperature (K) | 112 |
| *a*, *b*, *c* (Å) | 7.4776 (1), 19.1040 (2), 26.0919 (3) |
| *V* (Å$^3$) | 3727.28 (8) |
| *Z* | 8 |
| Radiation type | Mo *Kα* |
| $\mu$ (mm$^{-1}$) | 3.39 |
| Crystal size (mm) | 0.30 × 0.26 × 0.12 |
| | |
| Data collection | |
| Diffractometer | XtaLAB Synergy, Single source at offset/far, HyPix3000 |
| Absorption correction | Gaussian (*CrysAlis PRO*; Rigaku OD, 2019) |
| $T_{min}$, $T_{max}$ | 0.300, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 34066, 4062, 3774 |
| $R_{int}$ | 0.033 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.648 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.020, 0.044, 1.09 |
| No. of reflections | 4062 |
| No. of parameters | 242 |
| 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$^{-3}$) | 0.66, −0.92 |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2019), *SHELXT2014/5* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), and *OLEX2* (Dolomanov *et al.*, 2009).

(500 MHz, $CDCl_3$) $\delta$ 162.30, 161.11, 142.25, 141.81, 140.46, 128.32, 121.46, 120.15, 119.63, 79.69. MALDI–TOF MS: monoisotopic *m*/*z* calculated for $[M + H]^+$: 568.9; observed: 568.8.

## Refinement

Crystal data, data collection and structure refinement details of (I) are summarized in Table 2.


## Funding information

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# full crystallographic data

*IUCrData* (2022). **7**, x220895 [https://doi.org/10.1107/S2414314622008951]

## 2,2′-{(1*E*,1′*E*)-[Ethane-1,2-diylbis(azanylylidene)]bis(methanylylidene)}bis(4-iodophenol)

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**(I)**

*Crystal data*

$C_{20}H_{14}I_2N_2O_2$
$M_r$ = 568.13
Orthorhombic, *Pbca*
*a* = 7.4776 (1) Å
*b* = 19.1040 (2) Å
*c* = 26.0919 (3) Å
*V* = 3727.28 (8) Å$^3$
*Z* = 8
*F*(000) = 2160
$D_x$ = 2.025 Mg m$^{-3}$
Mo *Kα* radiation, $\lambda$ = 0.71073 Å
Cell parameters from 25879 reflections
$\theta$ = 1.5–27.4°
$\mu$ = 3.39 mm$^{-1}$
*T* = 112 K
Rect. Prism, orange
0.30 × 0.26 × 0.12 mm

*Data collection*

XtaLAB Synergy, Single source at offset/far, HyPix3000 diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2019)
$T_{min}$ = 0.300, $T_{max}$ = 1.000
34066 measured reflections
4062 independent reflections
3774 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033
$\theta_{max}$ = 27.4°, $\theta_{min}$ = 1.6°
*h* = −9→9
*k* = −23→24
*l* = −32→32

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.044
*S* = 1.09
4062 reflections
242 parameters
2 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0133P)^2 + 5.3948P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.66 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.92 e Å$^{-3}$
Extinction correction: SHELXL-2016/6 (Sheldrick 2015b, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.000120 (17)

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| I1 | 0.48524 (2) | 0.12444 (2) | 0.85138 (2) | 0.01709 (5) |
| I2 | 0.28304 (3) | 0.11138 (2) | 0.30070 (2) | 0.03584 (7) |
| O1 | 0.4758 (2) | 0.15083 (9) | 0.61326 (6) | 0.0205 (4) |
| O2 | 0.1200 (2) | 0.13607 (9) | 0.53361 (6) | 0.0233 (4) |
| N1 | 0.3343 (3) | 0.27475 (10) | 0.62428 (7) | 0.0162 (4) |
| N2 | 0.1963 (3) | 0.26760 (10) | 0.52825 (7) | 0.0163 (4) |
| C1 | 0.4718 (3) | 0.14643 (12) | 0.66467 (9) | 0.0159 (5) |
| C2 | 0.5299 (3) | 0.08468 (12) | 0.68798 (9) | 0.0183 (5) |
| H2A | 0.569184 | 0.046666 | 0.667387 | 0.022* |
| C3 | 0.5307 (3) | 0.07839 (12) | 0.74077 (9) | 0.0181 (5) |
| H3 | 0.571539 | 0.036324 | 0.756275 | 0.022* |
| C4 | 0.4721 (3) | 0.13336 (12) | 0.77109 (9) | 0.0158 (5) |
| C5 | 0.4097 (3) | 0.19424 (12) | 0.74922 (9) | 0.0162 (5) |
| H5 | 0.367238 | 0.231184 | 0.770347 | 0.019* |
| C6 | 0.4087 (3) | 0.20174 (12) | 0.69545 (8) | 0.0154 (5) |
| C7 | 0.3358 (3) | 0.26551 (12) | 0.67317 (8) | 0.0167 (5) |
| H7 | 0.288625 | 0.300786 | 0.694995 | 0.020* |
| C8 | 0.2667 (3) | 0.33732 (12) | 0.60266 (8) | 0.0152 (5) |
| C9 | 0.2710 (3) | 0.40210 (13) | 0.62775 (9) | 0.0190 (5) |
| H9 | 0.316488 | 0.405051 | 0.661683 | 0.023* |
| C10 | 0.2096 (3) | 0.46185 (12) | 0.60350 (9) | 0.0201 (5) |
| H10 | 0.212521 | 0.505533 | 0.620872 | 0.024* |
| C11 | 0.1437 (3) | 0.45812 (12) | 0.55393 (9) | 0.0199 (5) |
| H11 | 0.100415 | 0.499165 | 0.537540 | 0.024* |
| C12 | 0.1408 (3) | 0.39464 (12) | 0.52821 (9) | 0.0192 (5) |
| H12 | 0.095597 | 0.392368 | 0.494231 | 0.023* |
| C13 | 0.2040 (3) | 0.33413 (12) | 0.55195 (8) | 0.0154 (5) |
| C14 | 0.2275 (3) | 0.26073 (12) | 0.47991 (8) | 0.0161 (5) |
| H14 | 0.260311 | 0.300477 | 0.460144 | 0.019* |
| C15 | 0.2129 (3) | 0.19260 (12) | 0.45542 (8) | 0.0156 (5) |
| C16 | 0.1605 (3) | 0.13285 (12) | 0.48342 (9) | 0.0165 (5) |
| C17 | 0.1499 (3) | 0.06804 (12) | 0.45880 (9) | 0.0180 (5) |
| H17 | 0.115967 | 0.027658 | 0.477692 | 0.022* |
| C18 | 0.1884 (3) | 0.06224 (12) | 0.40725 (9) | 0.0186 (5) |
| H18 | 0.181371 | 0.018057 | 0.390663 | 0.022* |
| C19 | 0.2375 (3) | 0.12147 (12) | 0.37979 (9) | 0.0183 (5) |
| C20 | 0.2510 (3) | 0.18604 (12) | 0.40303 (8) | 0.0168 (5) |
| H20 | 0.285889 | 0.225866 | 0.383654 | 0.020* |
| H1 | 0.428 (3) | 0.1892 (8) | 0.6061 (10) | 0.020* |

| | | | | |
|---|---|---|---|---|
| H2 | 0.139 (3) | 0.1776 (7) | 0.5419 (9) | 0.020* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| I1 | 0.01938 (8) | 0.01833 (8) | 0.01356 (8) | −0.00151 (6) | −0.00173 (6) | 0.00283 (5) |
| I2 | 0.07494 (16) | 0.01979 (9) | 0.01279 (9) | 0.00557 (9) | 0.00604 (8) | −0.00114 (6) |
| O1 | 0.0249 (9) | 0.0219 (9) | 0.0145 (8) | 0.0034 (7) | 0.0003 (7) | −0.0015 (7) |
| O2 | 0.0358 (10) | 0.0204 (9) | 0.0136 (8) | −0.0054 (8) | 0.0043 (7) | 0.0029 (7) |
| N1 | 0.0167 (10) | 0.0177 (10) | 0.0142 (9) | −0.0021 (8) | −0.0008 (8) | 0.0004 (8) |
| N2 | 0.0172 (10) | 0.0182 (10) | 0.0135 (9) | −0.0003 (8) | −0.0012 (8) | −0.0005 (8) |
| C1 | 0.0121 (11) | 0.0218 (12) | 0.0138 (11) | −0.0023 (9) | −0.0012 (9) | −0.0008 (9) |
| C2 | 0.0162 (11) | 0.0177 (12) | 0.0211 (12) | 0.0006 (9) | 0.0006 (9) | −0.0046 (9) |
| C3 | 0.0149 (11) | 0.0180 (12) | 0.0215 (12) | 0.0000 (9) | −0.0022 (9) | 0.0022 (10) |
| C4 | 0.0161 (11) | 0.0187 (11) | 0.0127 (11) | −0.0035 (9) | −0.0009 (9) | 0.0001 (9) |
| C5 | 0.0167 (11) | 0.0181 (11) | 0.0138 (10) | −0.0004 (10) | −0.0006 (9) | −0.0010 (9) |
| C6 | 0.0145 (11) | 0.0170 (11) | 0.0148 (11) | −0.0020 (9) | −0.0013 (9) | −0.0004 (9) |
| C7 | 0.0180 (11) | 0.0171 (11) | 0.0150 (11) | −0.0018 (9) | −0.0005 (9) | −0.0033 (9) |
| C8 | 0.0152 (11) | 0.0155 (11) | 0.0149 (11) | 0.0001 (9) | 0.0018 (9) | 0.0008 (9) |
| C9 | 0.0203 (12) | 0.0209 (12) | 0.0157 (11) | −0.0029 (10) | 0.0003 (9) | −0.0025 (10) |
| C10 | 0.0192 (12) | 0.0163 (11) | 0.0248 (12) | −0.0009 (10) | 0.0035 (10) | −0.0042 (10) |
| C11 | 0.0195 (12) | 0.0161 (11) | 0.0241 (12) | 0.0030 (10) | 0.0025 (10) | 0.0041 (10) |
| C12 | 0.0205 (12) | 0.0206 (12) | 0.0165 (11) | 0.0016 (10) | −0.0017 (10) | 0.0020 (9) |
| C13 | 0.0148 (11) | 0.0165 (11) | 0.0149 (11) | −0.0004 (9) | 0.0020 (9) | 0.0010 (9) |
| C14 | 0.0163 (11) | 0.0170 (11) | 0.0150 (11) | −0.0010 (9) | −0.0016 (9) | 0.0023 (9) |
| C15 | 0.0147 (11) | 0.0186 (11) | 0.0135 (11) | −0.0010 (9) | −0.0017 (9) | 0.0006 (9) |
| C16 | 0.0165 (11) | 0.0193 (12) | 0.0136 (11) | −0.0004 (9) | −0.0006 (9) | 0.0029 (9) |
| C17 | 0.0196 (12) | 0.0165 (11) | 0.0179 (11) | −0.0027 (10) | −0.0009 (10) | 0.0036 (9) |
| C18 | 0.0202 (12) | 0.0157 (11) | 0.0199 (12) | −0.0005 (9) | −0.0027 (10) | −0.0017 (9) |
| C19 | 0.0244 (12) | 0.0188 (12) | 0.0117 (11) | 0.0023 (10) | 0.0005 (9) | −0.0001 (9) |
| C20 | 0.0215 (12) | 0.0151 (11) | 0.0138 (11) | −0.0014 (9) | 0.0009 (9) | 0.0015 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| I1—C4 | 2.104 (2) | C8—C9 | 1.401 (3) |
| I2—C19 | 2.100 (2) | C8—C13 | 1.405 (3) |
| O1—C1 | 1.344 (3) | C9—H9 | 0.9500 |
| O1—H1 | 0.835 (10) | C9—C10 | 1.384 (3) |
| O2—C16 | 1.345 (3) | C10—H10 | 0.9500 |
| O2—H2 | 0.836 (10) | C10—C11 | 1.386 (3) |
| N1—C7 | 1.288 (3) | C11—H11 | 0.9500 |
| N1—C8 | 1.415 (3) | C11—C12 | 1.386 (3) |
| N2—C13 | 1.415 (3) | C12—H12 | 0.9500 |
| N2—C14 | 1.290 (3) | C12—C13 | 1.394 (3) |
| C1—C2 | 1.397 (3) | C14—H14 | 0.9500 |
| C1—C6 | 1.409 (3) | C14—C15 | 1.454 (3) |
| C2—H2A | 0.9500 | C15—C16 | 1.411 (3) |
| C2—C3 | 1.383 (3) | C15—C20 | 1.402 (3) |

| | | | |
|---|---|---|---|
| C3—H3 | 0.9500 | C16—C17 | 1.397 (3) |
| C3—C4 | 1.386 (3) | C17—H17 | 0.9500 |
| C4—C5 | 1.377 (3) | C17—C18 | 1.380 (3) |
| C5—H5 | 0.9500 | C18—H18 | 0.9500 |
| C5—C6 | 1.410 (3) | C18—C19 | 1.389 (3) |
| C6—C7 | 1.456 (3) | C19—C20 | 1.378 (3) |
| C7—H7 | 0.9500 | C20—H20 | 0.9500 |

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| C1—O1—H1 | 105.6 (18) | C9—C10—C11 | 120.2 (2) |
| C16—O2—H2 | 104.9 (18) | C11—C10—H10 | 119.9 |
| C7—N1—C8 | 120.9 (2) | C10—C11—H11 | 119.9 |
| C14—N2—C13 | 120.8 (2) | C10—C11—C12 | 120.1 (2) |
| O1—C1—C2 | 118.7 (2) | C12—C11—H11 | 119.9 |
| O1—C1—C6 | 122.0 (2) | C11—C12—H12 | 119.8 |
| C2—C1—C6 | 119.3 (2) | C11—C12—C13 | 120.3 (2) |
| C1—C2—H2A | 119.7 | C13—C12—H12 | 119.8 |
| C3—C2—C1 | 120.6 (2) | C8—C13—N2 | 117.7 (2) |
| C3—C2—H2A | 119.7 | C12—C13—N2 | 122.5 (2) |
| C2—C3—H3 | 120.0 | C12—C13—C8 | 119.7 (2) |
| C2—C3—C4 | 120.1 (2) | N2—C14—H14 | 119.8 |
| C4—C3—H3 | 120.0 | N2—C14—C15 | 120.5 (2) |
| C3—C4—I1 | 119.49 (17) | C15—C14—H14 | 119.8 |
| C5—C4—I1 | 119.79 (17) | C16—C15—C14 | 121.2 (2) |
| C5—C4—C3 | 120.7 (2) | C20—C15—C14 | 119.6 (2) |
| C4—C5—H5 | 120.0 | C20—C15—C16 | 119.3 (2) |
| C4—C5—C6 | 120.0 (2) | O2—C16—C15 | 122.0 (2) |
| C6—C5—H5 | 120.0 | O2—C16—C17 | 118.4 (2) |
| C1—C6—C5 | 119.3 (2) | C17—C16—C15 | 119.6 (2) |
| C1—C6—C7 | 121.7 (2) | C16—C17—H17 | 119.8 |
| C5—C6—C7 | 119.0 (2) | C18—C17—C16 | 120.5 (2) |
| N1—C7—C6 | 120.9 (2) | C18—C17—H17 | 119.8 |
| N1—C7—H7 | 119.6 | C17—C18—H18 | 120.2 |
| C6—C7—H7 | 119.6 | C17—C18—C19 | 119.5 (2) |
| C9—C8—N1 | 123.5 (2) | C19—C18—H18 | 120.2 |
| C9—C8—C13 | 119.1 (2) | C18—C19—I2 | 118.35 (17) |
| C13—C8—N1 | 117.3 (2) | C20—C19—I2 | 120.16 (17) |
| C8—C9—H9 | 119.7 | C20—C19—C18 | 121.5 (2) |
| C10—C9—C8 | 120.5 (2) | C15—C20—H20 | 120.2 |
| C10—C9—H9 | 119.7 | C19—C20—C15 | 119.6 (2) |
| C9—C10—H10 | 119.9 | C19—C20—H20 | 120.2 |

| | | | |
|---|---|---|---|
| I1—C4—C5—C6 | 176.96 (17) | C8—N1—C7—C6 | −178.7 (2) |
| I2—C19—C20—C15 | 177.30 (17) | C8—C9—C10—C11 | −0.3 (4) |
| O1—C1—C2—C3 | 178.9 (2) | C9—C8—C13—N2 | −178.1 (2) |
| O1—C1—C6—C5 | −179.3 (2) | C9—C8—C13—C12 | −2.5 (3) |
| O1—C1—C6—C7 | 3.2 (3) | C9—C10—C11—C12 | −0.7 (4) |
| O2—C16—C17—C18 | 179.4 (2) | C10—C11—C12—C13 | 0.0 (4) |
| N1—C8—C9—C10 | 177.2 (2) | C11—C12—C13—N2 | 177.0 (2) |

| | | | |
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| N1—C8—C13—N2 | 6.2 (3) | C11—C12—C13—C8 | 1.6 (3) |
| N1—C8—C13—C12 | −178.1 (2) | C13—N2—C14—C15 | −177.7 (2) |
| N2—C14—C15—C16 | 1.5 (3) | C13—C8—C9—C10 | 1.8 (3) |
| N2—C14—C15—C20 | −178.7 (2) | C14—N2—C13—C8 | −145.5 (2) |
| C1—C2—C3—C4 | 0.6 (4) | C14—N2—C13—C12 | 38.9 (3) |
| C1—C6—C7—N1 | −3.3 (3) | C14—C15—C16—O2 | 0.7 (4) |
| C2—C1—C6—C5 | 1.6 (3) | C14—C15—C16—C17 | −179.2 (2) |
| C2—C1—C6—C7 | −176.0 (2) | C14—C15—C20—C19 | 179.9 (2) |
| C2—C3—C4—I1 | −177.29 (17) | C15—C16—C17—C18 | −0.7 (4) |
| C2—C3—C4—C5 | 1.1 (3) | C16—C15—C20—C19 | −0.3 (3) |
| C3—C4—C5—C6 | −1.5 (3) | C16—C17—C18—C19 | −0.2 (4) |
| C4—C5—C6—C1 | 0.1 (3) | C17—C18—C19—I2 | −177.10 (17) |
| C4—C5—C6—C7 | 177.7 (2) | C17—C18—C19—C20 | 0.8 (4) |
| C5—C6—C7—N1 | 179.2 (2) | C18—C19—C20—C15 | −0.6 (4) |
| C6—C1—C2—C3 | −1.9 (3) | C20—C15—C16—O2 | −179.1 (2) |
| C7—N1—C8—C9 | 29.0 (3) | C20—C15—C16—C17 | 1.0 (3) |
| C7—N1—C8—C13 | −155.5 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2*A*···I2$^{i}$ | 0.95 | 3.32 | 4.009 (2) | 131 |
| C5—H5···I2$^{ii}$ | 0.95 | 3.17 | 4.061 (2) | 156 |
| C7—H7···I2$^{ii}$ | 0.95 | 3.23 | 4.094 (2) | 152 |
| C18—H18···I1$^{iii}$ | 0.95 | 3.16 | 4.066 (2) | 159 |
| O1—H1···N1 | 0.84 (1) | 1.84 (2) | 2.609 (3) | 152 (3) |
| O2—H2···N2 | 0.84 (1) | 1.81 (2) | 2.580 (3) | 153 (3) |

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