



# Consistent supramolecular motif of $C(7)$ $O-H \cdots O$ hydrogen-bonded chains and different local symmetries in three isoindole-4-carboxylic acid derivatives

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Received 20 November 2025

Accepted 1 December 2025

Edited by W. T. A. Harrison, University of Aberdeen, United Kingdom

**Keywords:** crystal structure; hydrogen bonds; zigzag chains; Hirshfeld surface analysis.

**CCDC references:** 2512457; 2512456; 2512455

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

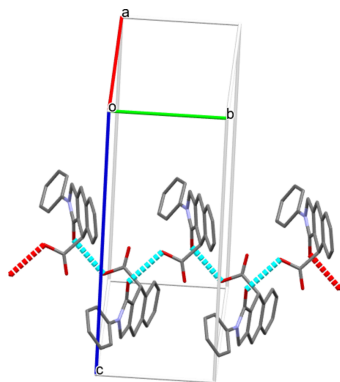
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The syntheses and structures of (3*aRS*,4*RS*,9*aSR*)-3-oxo-2-(2-phenylethyl)-2,3,3*a*,4,9,9*a*-hexahydro-1*H*-benzo[*f*]isoindole-4-carboxylic acid,  $C_{21}H_{21}NO_3$  (I), (3*aRS*,4*RS*,9*aSR*)-3-oxo-2-(propan-2-yl)-2,3,3*a*,4,9,9*a*-hexahydro-1*H*-benzo[*f*]isoindole-4-carboxylic acid,  $C_{16}H_{19}NO_3$  (II) and (4*RS*)-3-oxo-2-phenyl-2,3,4,9-tetrahydro-1*H*-benzo[*f*]isoindole-4-carboxylic acid,  $C_{19}H_{15}NO_3$  (III), are described. Compound (I) crystallizes with two molecules in the asymmetric unit. In the extended structures of (I), (II) and (III) the molecules are linked by  $O_c-H \cdots O_i$  ( $c$  = carboxylic acid,  $i$  = indole) hydrogen bonds, forming a common  $C(7)$  zigzag chain motif propagating along the [010] direction, despite the different local symmetries. These chains are connected by weak  $C-H \cdots O$  and  $C-H \cdots \pi$  interactions, forming a three-dimensional network in each case. A Hirshfeld surface analysis was undertaken to investigate and quantify the intermolecular interactions, which are dominated by  $H \cdots H$ ,  $C \cdots H/H \cdots C$  and  $O \cdots H/H \cdots O$  contacts in each case.

## 1. Chemical context

The isoindole motif is a common structural element found in numerous natural and biologically active compounds. This fragment occurs in cyclopiazonic acid, cytochalasan and anthraquinone alkaloids, as well as in meroterpenoids of the *Stachybotrys* species (Speck & Magauer, 2013). Compounds containing the isoindole skeleton exhibit a wide range of biological activities, including anticancer and antimicrobial properties (Bhatia, 2016).

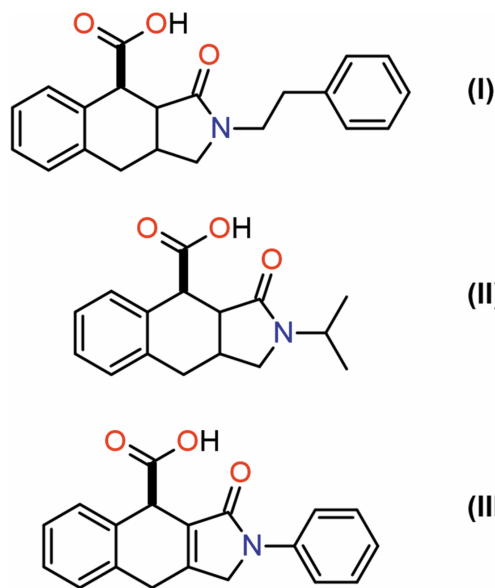
Among the various synthetic approaches used to construct the isoindole core, the intramolecular Diels–Alder reaction of vinylarenes (IMDAV) has emerged as a particularly powerful method (see the recent review by Krishna *et al.*, 2022). This reaction, as an extension of the traditional Diels–Alder methodology, provides an efficient route to complex polycyclic aromatic and carbocyclic systems. In the IMDAV reaction, the vinylarene moiety acts as the dienophile, enabling the formation of fused or bridged ring structures with remarkable regio- and stereocontrol. By positioning the reactive partners within the same molecule, the IMDAV process overcomes the typically low reactivity of vinylarenes observed in intermolecular Diels–Alder reactions, thereby enhancing both reaction rates and selectivity. This transformation is especially valuable for constructing the isoindole scaffold, including partially hydrogenated isoindoles fused with benzene



(Voronov *et al.*, 2018), furan (Fischer & Hünig, 1987; Alekseeva *et al.*, 2020), or thiophene (Herz *et al.*, 2001; Nadirova *et al.*, 2020) rings.

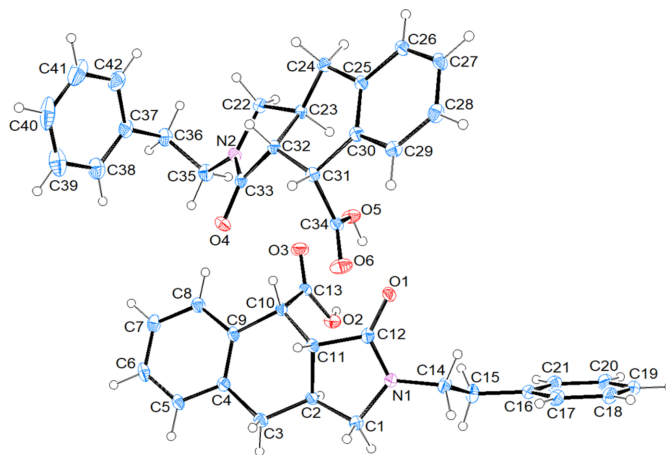
The significance of the IMDAV reaction lies in its broad utility for the synthesis of natural products, pharmaceuticals, and advanced materials. Many biologically active compounds contain fused aromatic frameworks or rigid polycyclic skeletons that can be efficiently accessed through IMDAV strategies. Furthermore, the ability to incorporate aromatic functionality directly into the newly formed ring systems provides unique opportunities for subsequent functionalization and molecular diversification. It is noteworthy that the target isoindoles described in this work are structurally related to compounds exhibiting selective inhibitory activity toward PTP (De Cesco *et al.*, 2012) and PARP enzymes (Papeo *et al.*, 2015).

In this study, we report the syntheses and structures of three new isoindole derivatives (3*aRS*,4*RS*,9*aSR*)-3-oxo-2-(2-phenylethyl)-2,3,3*a*,4,9,9*a*-hexahydro-1*H*-benzo[*f*]isoindole-4-carboxylic acid, C<sub>21</sub>H<sub>21</sub>NO<sub>3</sub> (I), (3*aRS*,4*RS*, 9*aSR*)-3-oxo-2-(propan-2-yl)-2,3,3*a*,4,9,9*a*-hexahydro-1*H*-benzo[*f*]isoindole-4-carboxylic acid, C<sub>16</sub>H<sub>19</sub>NO<sub>3</sub> (II) and (4*RS*)-3-oxo-2-phenyl-2,3,4,9-tetrahydro-1*H*-benzo[*f*]isoindole-4-carboxylic acid, C<sub>19</sub>H<sub>15</sub>NO<sub>3</sub> (III). Compounds (I)–(III) are obtainable from readily available precursors – secondary amines and maleic anhydrides. The proposed synthetic approach does not require strictly anhydrous solvents or inert atmospheres and enables a practically one-pot, diastereoselective formation of complex polycyclic systems.



## 2. Structural commentary

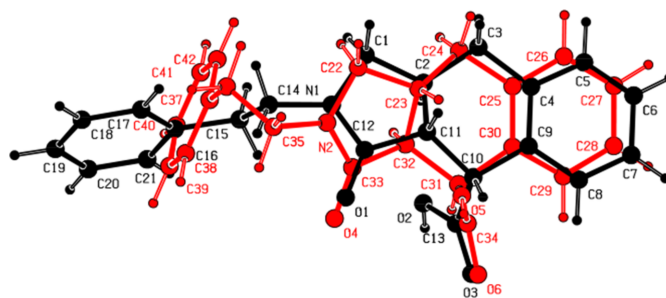
The asymmetric unit of compound (I) (space group  $P2_1/n$ ) consists of two molecules (*A* containing N1 and *B* containing N2) (Fig. 1). Each molecule contains three stereogenic (chiral) centres: in the arbitrarily chosen asymmetric unit, C2, C10 and C11 have *S*, *R* and *R* configurations, respectively, in molecule



**Figure 1**

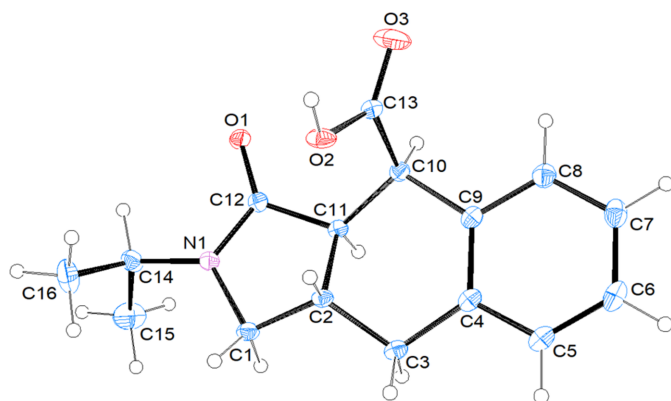
The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level.

*A* and C23, C31 and C32 have *S*, *R* and *R* configurations, respectively, in molecule *B*. In molecule *A* (Fig. 1), the least-squares plane (r.m.s. deviation = 0.002 Å) of the thirteen-membered ring system (N1/C1–C12) indicates near planarity and it makes a dihedral angle of 75.11 (10)<sup>o</sup> with the terminal phenyl ring (C16–C21). The 2,5-dihydro-1*H*-pyrrole ring (N1/C1/C2/C11/C12) adopts an envelope conformation on atom C2 with puckering parameters (Cremer & Pople, 1975)  $Q(2) = 0.344$  (3) Å,  $\varphi(2) = 258.4$  (4)<sup>o</sup> and the cyclohexa-1,4-diene ring (C2–C4/C9–C11) exhibits a twist-boat conformation [the puckering parameters are  $Q_T = 0.508$  (3) Å,  $\theta = 127.6$  (3)<sup>o</sup>,  $\varphi = 161.9$  (4)<sup>o</sup>]. In molecule *B* (Fig. 1), the best plane (r.m.s. deviation = 0.002 Å) of the thirteen-membered ring system (N2/C22–C33) makes a dihedral angle of 80.86 (10)<sup>o</sup> with the terminal phenyl ring (C37–C42). The 2,5-dihydro-1*H*-pyrrole ring (N2/C22/C23/C32/C33) adopts an envelope conformation on atom C23 with puckering parameters of  $Q(2) = 0.356$  (2) Å,  $\varphi(2) = 253.2$  (4)<sup>o</sup>, and the cyclohexa-1,4-diene ring (C23–C25/C30–C32) exhibits a twist-boat conformation with puckering parameters  $Q_T = 0.523$  (2) Å,  $\theta = 128.8$  (2)<sup>o</sup> and  $\varphi = 164.9$  (3)<sup>o</sup>. The major difference between *A* and *B* is seen in the N<sub>i</sub>–C–C–C<sub>ar</sub> (*i* = indole, ar = aromatic) torsion angles, being 171.36 (19)<sup>o</sup> (*anti*) for molecule *A* and –61.4 (3)<sup>o</sup> (*gauche*) for *B*. An overlay fit of the inverted molecule *B* on



**Figure 2**

A least-squares overlay of the two independent molecules (*A* black and *B* red) in (I).

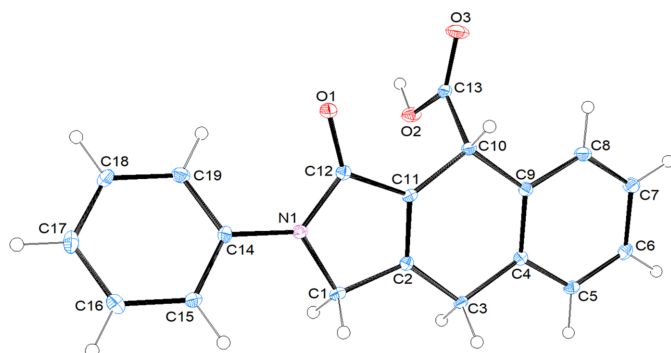


**Figure 3**  
The molecular structure of (II), showing displacement ellipsoids drawn at the 50% probability level.

molecule *A* is shown in Fig. 2 with the weighted r.m.s. fit of the 25 non-H atoms being 2.34 Å.

Compound (II) crystallizes in space group *C2/c* with one molecule in the asymmetric unit (Fig. 3). The stereogenic centres in the arbitrarily chosen asymmetric molecule – C2, C10 and C11 – have *S*, *R* and *R* configurations, respectively, but crystal symmetry generates a racemic mixture. The thirteen-membered ring system (N1/C1–C12) is essentially planar (r.m.s. deviation = 0.002 Å). The 2,5-dihydro-1*H*-pyrrole ring (N1/C1/C2/C11/C12) adopts an envelope conformation on atom C2 with puckering parameters of  $Q(2) = 0.3634(17)$  Å and  $\varphi(2) = 255.1(3)^\circ$ , and the cyclohexa-1,4-diene ring (C2–C4/C9–C11) has a twist-boat conformation [the puckering parameters are  $Q_T = 0.4941(17)$  Å,  $\theta = 132.0(2)^\circ$ ,  $\varphi = 157.7(3)^\circ$ ].

In (III), which crystallizes in space group *P2<sub>1</sub>/c* (Fig. 4), the thirteen-membered ring system (N1/C1–C12) is essentially planar (r.m.s. deviation = 0.002 Å) and makes a dihedral angle of 32.15 (9)° with the terminal phenyl ring (C14–C19). The sole stereogenic centre, C10, has an *R* configuration in the arbitrarily chosen asymmetric unit, but crystal symmetry generates a racemic mixture. The 2,5-dihydro-1*H*-pyrrole ring (N1/C1/C2/C11/C12) and the cyclohexa-1,4-diene ring (C2–C4/C9–C11) are essentially planar with maximum deviations



**Figure 4**  
The molecular structure of (III), showing displacement ellipsoids drawn at the 50% probability level.

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

*Cg3*, *Cg4*, *Cg10* and *Cg11* are the centroids of the C4–C9, C16–C21, C25–C30 and C37–C42 rings, respectively.

| <i>D</i> –H... <i>A</i>      | <i>D</i> –H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> –H... <i>A</i> |
|------------------------------|-------------|---------------|-----------------------|-------------------------|
| O2–H2O...O4 <sup>i</sup>     | 0.85        | 1.77          | 2.607 (2)             | 166                     |
| O5–H5O...O1                  | 0.85        | 1.76          | 2.606 (2)             | 171                     |
| C2–H2...O2                   | 1.00        | 2.38          | 3.030 (3)             | 122                     |
| C15–H15A...O6 <sup>i</sup>   | 0.99        | 2.52          | 3.404 (3)             | 148                     |
| C23–H23...O5                 | 1.00        | 2.34          | 3.018 (3)             | 124                     |
| C3–H3A...Cg3 <sup>ii</sup>   | 0.99        | 2.75          | 3.702 (3)             | 161                     |
| C7–H7...Cg11 <sup>iii</sup>  | 0.95        | 2.86          | 3.796 (3)             | 167                     |
| C20–H20...Cg4 <sup>iv</sup>  | 0.95        | 2.95          | 3.783 (3)             | 148                     |
| C24–H24A...Cg10 <sup>v</sup> | 0.99        | 2.71          | 3.648 (3)             | 158                     |

Symmetry codes: (i)  $x, y + 1, z$ ; (ii)  $-x, -y + 1, -z + 1$ ; (iii)  $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$ ; (iv)  $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$ ; (v)  $-x + 1, -y, -z + 1$ .

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

*Cg3* is the centroid of the C4–C9 rings.

| <i>D</i> –H... <i>A</i>     | <i>D</i> –H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> –H... <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| O2–H2O...O1 <sup>i</sup>    | 0.86        | 1.74          | 2.5885 (17)           | 171                     |
| C2–H2...O2                  | 1.00        | 2.33          | 3.002 (2)             | 124                     |
| C3–H3B...O2 <sup>ii</sup>   | 0.99        | 2.56          | 3.500 (2)             | 159                     |
| C16–H16A...O3 <sup>i</sup>  | 0.98        | 2.55          | 3.531 (3)             | 176                     |
| C3–H3A...Cg3 <sup>iii</sup> | 0.99        | 2.68          | 3.6164 (16)           | 158                     |

Symmetry codes: (i)  $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$ ; (ii)  $-x + \frac{3}{2}, -y + \frac{3}{2}, -z + 1$ ; (iii)  $-x + \frac{3}{2}, -y + \frac{1}{2}, -z + 1$ .

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

*Cg1*, *Cg2*, *Cg3* and *Cg4* are the centroids of the N1/C1/C2/C11/C12, C2–C11 C4–C9 and C16–C21 rings, respectively.

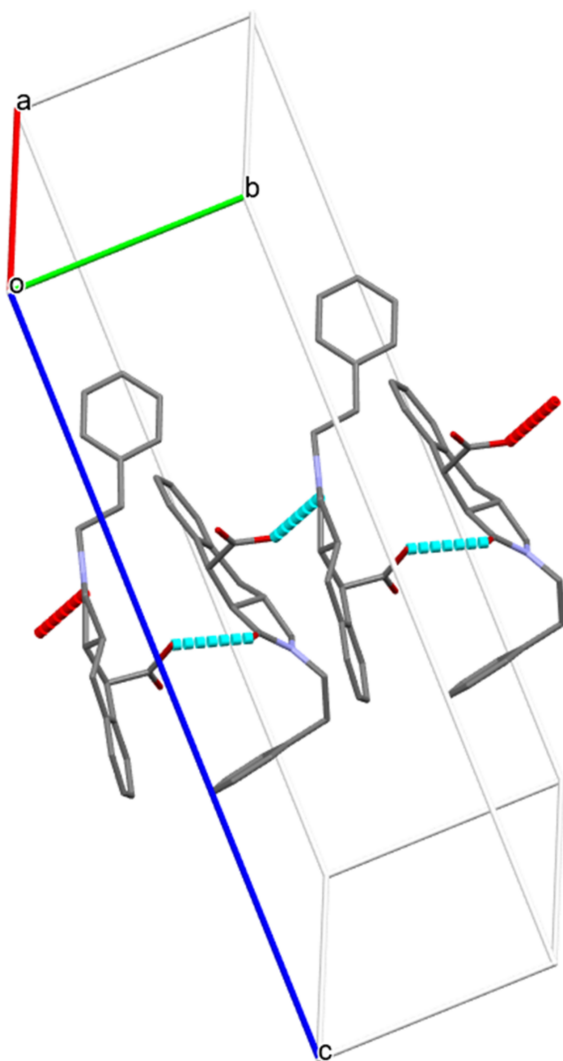
| <i>D</i> –H... <i>A</i>     | <i>D</i> –H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> –H... <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| O2–H2O...O1 <sup>i</sup>    | 0.84        | 1.90          | 2.741 (2)             | 174                     |
| C1–H1A...Cg2 <sup>ii</sup>  | 0.99        | 2.91          | 3.756 (2)             | 144                     |
| C1–H1B...Cg3 <sup>iii</sup> | 0.99        | 2.58          | 3.427 (2)             | 144                     |
| C3–H3A...Cg1 <sup>ii</sup>  | 0.99        | 2.70          | 3.593 (2)             | 150                     |
| C3–H3B...Cg2 <sup>iii</sup> | 0.99        | 2.60          | 3.388 (2)             | 137                     |
| C18–H18...Cg4 <sup>iv</sup> | 0.95        | 2.61          | 3.452 (2)             | 148                     |

Symmetry codes: (i)  $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$ ; (ii)  $-x + 1, -y + 1, -z + 1$ ; (iii)  $-x + 1, -y + 2, -z + 1$ ; (iv)  $-x + 2, y - \frac{1}{2}, -z + \frac{3}{2}$ .

of  $-0.023(2)$  Å for N1 and  $0.012(2)$  Å for C9, respectively. Otherwise, the bond lengths and angles in the molecules of (I), (II) and (III) are comparable with each other and with those of related structures detailed in the *Database survey* (section 4).

### 3. Supramolecular features and Hirshfeld surface analysis

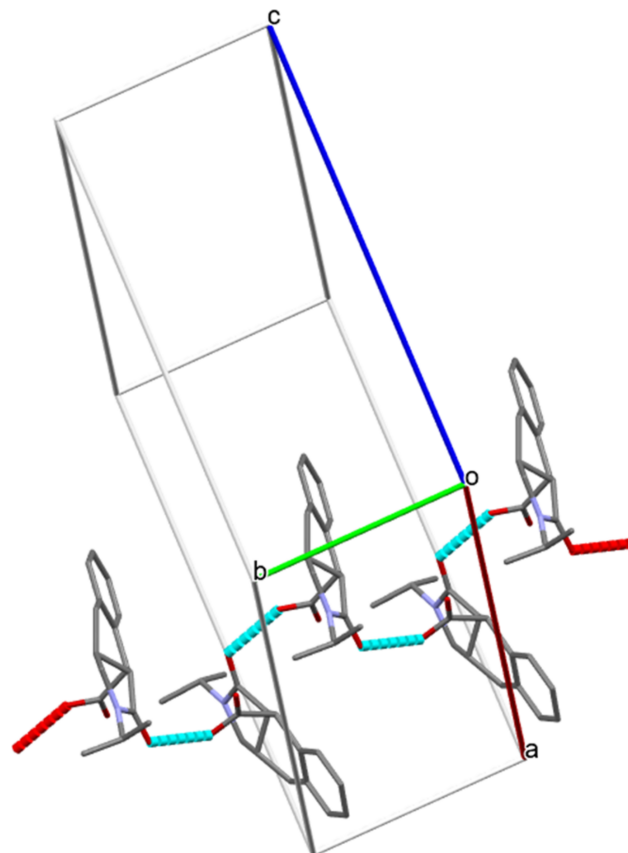
In the extended structures of (I), (II), and (III), the molecules are linked by a consistent O–H...O hydrogen bond from the carboxylic acid OH group to the indole O atom, forming a *C*(7) zigzag chain propagating along the [010] direction in each case (Tables 1, 2 and 3; Figs. 5, 6 and 7). Thus, it may be noted that carboxylic acid homodimers linked by pairs of O–H...O hydrogen bonds are *not* formed. In (I), the *A* and *B* molecules alternate in the chain and alternate molecules of each kind are related by simple unit-cell translation in the *b*-axis direction.


**Figure 5**

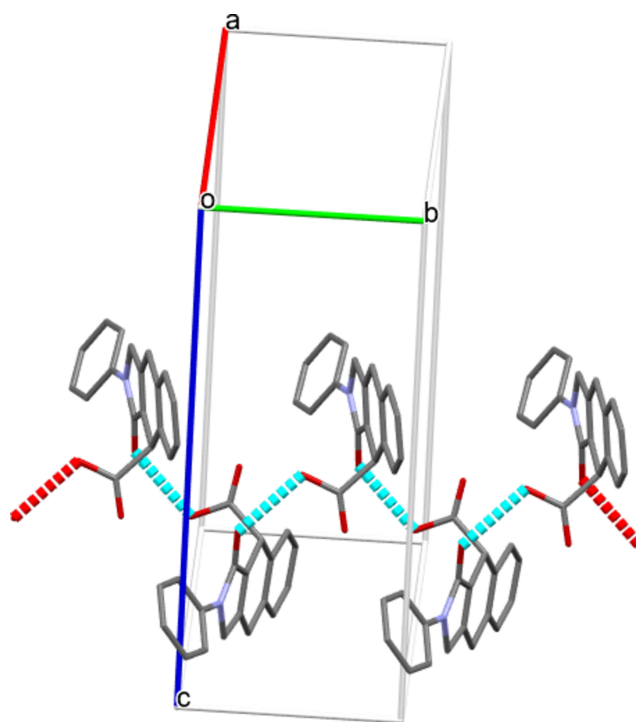
Partial packing diagram for (I) showing O—H...O hydrogen bonds forming C(7) zigzag chains propagating along the [010] direction. The remaining hydrogen atoms are omitted for clarity.

In (II) and (III), adjacent molecules in the chain are related by a  $2_1$  screw axis. These chains are connected by various C—H...O and C—H... $\pi$  interactions, forming three-dimensional networks in each case (Figs. S1–S9 in the supporting information).

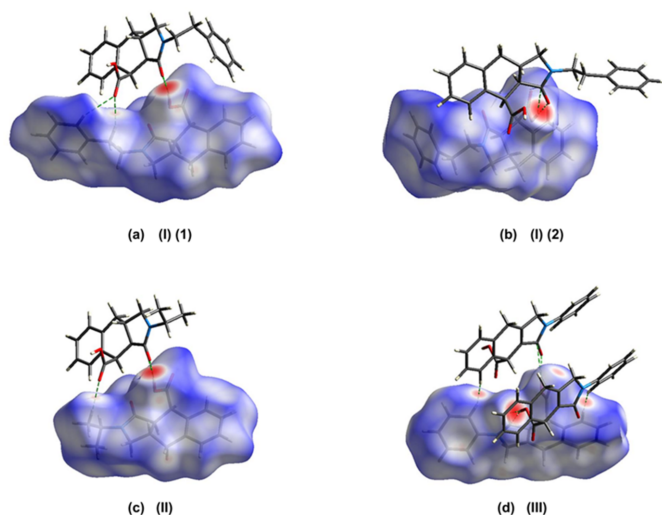
The Hirshfeld surfaces and corresponding two-dimensional fingerprint plots were calculated using *Crystal Explorer 17.5* (Spackman *et al.*, 2021) to further quantify the intermolecular interactions. The  $d_{\text{norm}}$  mappings for molecules (I)A, (I)B, (II) and (III) were performed in the ranges  $-0.76$  to  $+1.46$  a.u.,  $-0.76$  to  $+1.46$  a.u.,  $-0.78$  to  $+1.41$  a.u. and  $-0.66$  to  $+1.21$  a.u., respectively. The O—H...O and C—H...O interactions are indicated by red areas on the Hirshfeld surfaces; see Fig. 8a for (I)A, Fig. 8b for (I)B, Fig. 8c for (II), and Fig. 8d for (III). The two-dimensional fingerprint plots are given in Fig. 9, where H...H, C...H/H...C and O...H/H...O interactions dominate for all three structures with the other contact types making a negligible contribution (Table 4).


**Figure 6**

Partial packing diagram for (II) showing O—H...O hydrogen bonds forming C(7) zigzag chains propagating along the [010] direction. The remaining hydrogen atoms are omitted for clarity.


**Figure 7**

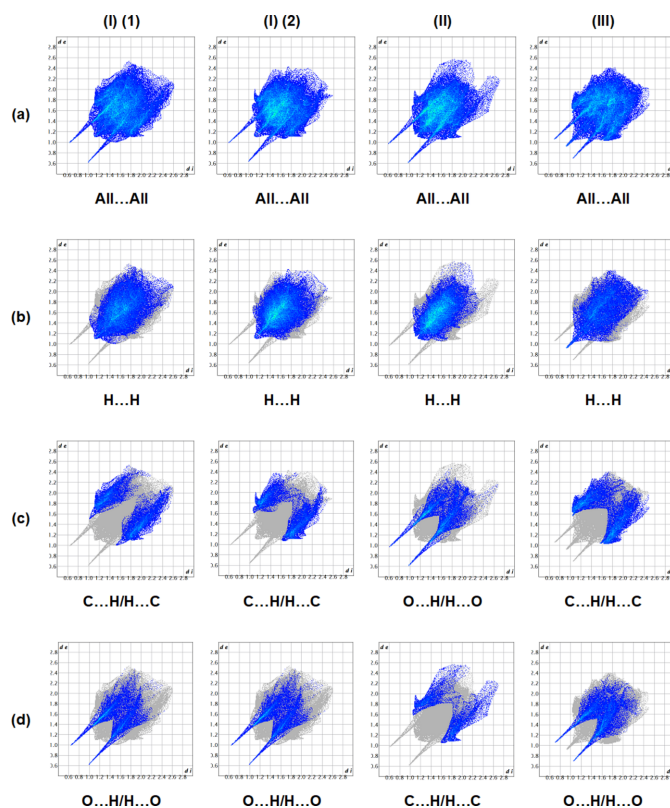
Partial packing diagram for (III) showing O—H...O hydrogen bonds forming C(7) zigzag chains propagating along the [010] direction. The remaining hydrogen atoms are omitted for clarity.



**Figure 8**  
Views of the three-dimensional Hirshfeld surfaces of the compounds (I) *A*, (I) *B*, (II) and (III) mapped over  $d_{\text{norm}}$ .

#### 4. Database survey

A Cambridge Structural Database (CSD, Version 6.00, last update April 2025; Groom *et al.*, 2016) search indicated that



**Figure 9**  
A view of the two-dimensional fingerprint plots for compounds (I) *A*, (I) *B*, (II) and (III), showing (a) all interactions, and delineated into (b)  $\text{H}\cdots\text{H}$ , (c)  $\text{C}\cdots\text{H}/\text{H}\cdots\text{C}$  for (I) *A*, (I) *B* and (III), and  $\text{O}\cdots\text{H}/\text{H}\cdots\text{O}$  for (II) and (d)  $\text{O}\cdots\text{H}/\text{H}\cdots\text{O}$  for (I) *A*, (I) *B* and (III), and  $\text{C}\cdots\text{H}/\text{H}\cdots\text{C}$  for (II) interactions.

**Table 4**

The Hirshfeld fingerprint contact percentages (%) for (I), (II) and (III).

| Contacts  | (I) <i>A</i> | (I) <i>B</i> | (II) | (III) |
|---|--------------|--------------|------|-------|
| $\text{H}\cdots\text{H}$                        | 53.7         | 59.8         | 58.0 | 39.2  |
| $\text{C}\cdots\text{H}/\text{H}\cdots\text{C}$ | 24.7         | 20.6         | 14.9 | 30.6  |
| $\text{O}\cdots\text{H}/\text{H}\cdots\text{O}$ | 20.2         | 18.1         | 26.7 | 25.8  |
| $\text{O}\cdots\text{C}/\text{C}\cdots\text{O}$ | 1.0          | 1.1          | 0.2  | 0.2   |
| $\text{O}\cdots\text{O}$                        | 0.3          | 0.2          | 0.2  | 0.3   |
| $\text{N}\cdots\text{H}/\text{H}\cdots\text{N}$ | 0.2          | 0.1          | 0.0  | 0.5   |
| $\text{N}\cdots\text{C}/\text{C}\cdots\text{N}$ | 0.0          | 0.0          | 0.0  | 0.3   |
| $\text{C}\cdots\text{C}$                        | 0.0          | 0.0          | 0.0  | 1.7   |

the five most similar compounds to the title compounds containing the 2,3,4,5,6,7-hexahydro-1*H*-isoindole unit are CSD refcodes EHURIM (Yakovleva *et al.*, 2025), OJIPUV (Zaytsev *et al.*, 2021), TODKEF (Elliott & Booker-Milburn, 2019), BAFYAL (Zhong *et al.*, 2017) and QAFSUO (Zubkov *et al.*, 2016). In the crystal of EHURIM, the molecules are connected by  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds, forming layers lying parallel to the (101) plane. Furthermore, the molecules form layers parallel to the (10 $\bar{2}$ ) plane by way of  $\text{C}-\text{H}\cdots\pi$  interactions. In OJIPUV the molecules are connected by  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds,  $\text{C}-\text{H}\cdots\pi$  interactions and  $\pi-\pi$  stacking interactions, forming a three-dimensional network. In TODKEF, the molecules are linked by  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds, forming a three-dimensional network with  $\text{C}-\text{H}\cdots\pi$  interactions also observed. In BAFYAL, the molecules are linked by  $\text{C}-\text{H}\cdots\text{O}$  interactions, forming layers lying parallel to the (002) plane;  $\pi-\pi$  interactions are also present. In QAFSUO, the three-dimensional packing is consolidated by  $\text{O}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  contacts and  $\text{C}-\text{H}\cdots\pi$  interactions.

#### 5. Synthesis and crystallization

To prepare (I), (2*E*)-3-phenyl-*N*-(2-phenylethyl)prop-2-en-1-amine (0.73 g, 3.06 mmol) was dissolved in 1,4-dioxane (15 ml) and maleic anhydride (0.30 g, 3.06 mmol) was added. The reaction mixture was stirred for 30 min; after that 4-(dimethylamino)pyridine (0.75 g, 6.12 mmol) was added. The resulting solution was refluxed for 8 h. After cooling to room temperature, the reaction mixture was poured into water (30 ml) and acidified with concentrated hydrochloric acid to pH  $\sim$ 6. The aqueous phase was extracted with ethyl acetate (3  $\times$  10 ml). The combined organic layers were dried over sodium sulfate, filtered, and concentrated under reduced pressure. Upon addition of ethyl acetate to the residue, a white precipitate formed, which was collected by filtration and air-dried to afford the target product as a white solid (0.53 g, 1.60 mmol, 52%, m.p. 510–513 K). Single crystals of (I) suitable for X-ray diffraction analysis were grown from the mixed solvents of ethanol and dimethylformamide.

$^1\text{H}$  NMR (600 MHz, DMSO- $d_6$ , 293 K) (*J*, Hz):  $\delta$  12.45 (*br. s*, 1H, COOH), 7.44–7.43 (*m*, 1H, H-Ar), 7.30–7.28 (*m*, 2H, H-Ar), 7.26–7.25 (*m*, 2H, H-Ar), 7.22–7.16 (*m*, 4H, H-Ar), 3.94 (*d*, *J* = 6.1, 1H, H-Ar), 3.49–3.45 (*m*, 1H, H-NCH<sub>2</sub>CH<sub>2</sub>), 3.42–3.37 (*m*, 2H, H-NCH<sub>2</sub>CH<sub>2</sub>, H-1A), 3.08 (*dd*, *J* = 9.1, 9.6, 1H, H-1), 2.99–2.91 (*m*, 2H, H-3a,9a), 2.80–2.73 (*m*, 2H, H-

**Table 5**  
Experimental details.

|   | (I)   | (II)  | (III)   |
|---|---|---|---|
| Crystal data  |   |   |   |
| Chemical formula  | C <sub>21</sub> H <sub>21</sub> NO <sub>3</sub>           | C <sub>16</sub> H <sub>19</sub> NO <sub>3</sub>           | C <sub>19</sub> H <sub>15</sub> NO <sub>3</sub>           |
| <i>M<sub>r</sub></i>  | 335.39  | 273.32  | 305.32  |
| Crystal system, space group   | Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>n</i>            | Monoclinic, <i>C</i> 2/ <i>c</i>                          | Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>c</i>            |
| Temperature (K)   | 150   | 150   | 100   |
| <i>a</i> , <i>b</i> , <i>c</i> (Å)  | 17.3659 (15), 8.0220 (7), 25.883 (2)                      | 25.1799 (12), 7.9085 (3),<br>17.6079 (8)                  | 11.9915 (10), 7.1296 (6),<br>16.7967 (16)                 |
| $\beta$ (°)   | 109.250 (4)   | 126.771 (2)   | 99.338 (5)  |
| <i>V</i> (Å <sup>3</sup> )  | 3404.2 (5)  | 2808.7 (2)  | 1417.0 (2)  |
| <i>Z</i>  | 8   | 8   | 4   |
| Radiation type  | Mo <i>K</i> $\alpha$                                      | Mo <i>K</i> $\alpha$                                      | Mo <i>K</i> $\alpha$                                      |
| $\mu$ (mm <sup>-1</sup> )   | 0.09  | 0.09  | 0.10  |
| Crystal size (mm)   | 0.26 × 0.24 × 0.21  | 0.31 × 0.29 × 0.29  | 0.40 × 0.04 × 0.03  |
| Data collection   |   |   |   |
| Diffractometer  | Bruker APEXII CCD   | Bruker APEXII CCD   | Bruker Kappa APEXII area-detector diffractometer          |
| Absorption correction   | Multi-scan ( <i>SADABS</i> ; Krause <i>et al.</i> , 2015) | Multi-scan ( <i>SADABS</i> ; Krause <i>et al.</i> , 2015) | Multi-scan ( <i>SADABS</i> ; Krause <i>et al.</i> , 2015) |
| <i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>   | 0.964, 0.973  | 0.964, 0.967  | 0.842, 1.000  |
| No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections                             | 46007, 6513, 3839   | 12572, 3240, 2416   | 13142, 3226, 1938   |
| <i>R</i> <sub>int</sub>   | 0.115   | 0.036   | 0.088   |
| ( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )   | 0.612   | 0.650   | 0.655   |
| Refinement  |   |   |   |
| <i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i> | 0.048, 0.118, 1.01  | 0.042, 0.108, 1.03  | 0.054, 0.128, 1.01  |
| No. of reflections  | 6513  | 3240  | 3226  |
| No. of parameters   | 453   | 184   | 209   |
| H-atom treatment  | H-atom parameters constrained                             | H-atom parameters constrained                             | H-atom parameters constrained                             |
| $\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )  | 0.25, -0.24   | 0.32, -0.19   | 0.31, -0.24   |

Computer programs: *APEX2* (Bruker, 2007), *APEX3* (Bruker, 2016), *SAINTE* (Bruker, 2007), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

NCH<sub>2</sub>CH<sub>2</sub>), 2.66 (*br. dd*, *J* = 12.6, 16.1, 1H, H-9A), 2.38 (*dd*, *J* = 5.5, 12.6, 1H, H-9B) ppm. <sup>13</sup>C {<sup>1</sup>H} NMR (151 MHz, DMSO-*d*<sub>6</sub>, 296 K)  $\delta$  173.0, 172.8, 139.2, 136.6, 133.0, 130.0, 129.9, 128.7 (2C), 128.4 (2C), 127.0, 126.2, 126.1, 51.0, 46.1, 43.6, 42.4, 33.4, 32.8, 32.0 ppm. IR (KBr),  $\nu$  (cm<sup>-1</sup>) 3011 (COOH), 1715 (C=O), 1640 (NCO). Analysis calculated for C<sub>21</sub>H<sub>21</sub>NO<sub>3</sub>: C, 75.20; H, 6.31; N, 4.18. Found: C, 75.07; H, 6.44; N, 4.39.

To prepare (II), (2*E*)-3-phenyl-*N*-(propan-2-yl)prop-2-en-1-amine (0.53 g, 3.06 mmol) was dissolved in acetonitrile (15 ml), and maleic anhydride (0.30 g, 3.06 mmol) was added. The reaction mixture was refluxed for 8 h. Upon slowly cooling to room temperature, the product crystallized from the reaction mixture. The resulting solid was collected by filtration, washed with diethyl ether, and air-dried to afford the target compound as a white crystalline solid (0.57 g, 2.08 mmol, 68%, m.p. 502–503 K). A single crystal of (II) suitable for X-ray diffraction analysis was isolated from the obtained crystalline material.

<sup>1</sup>H NMR (600 MHz, DMSO-*d*<sub>6</sub>, 295 K) (*J*, Hz):  $\delta$  12.37 (*br. s*, 1H, COOH), 7.43 (*dd*, *J* = 2.1, 7.5, 1H, H-5-Ph), 7.21–7.17 (*m*, 3H, H-6,7,8-Ph), 4.17 (*hept*, *J* = 6.8, 1H, H-2-*i*-Pr), 3.93 (*dd*, *J* = 6.1, 1H, H-4), 3.55 (*dd*, *J* = 6.6, 8.6, 1H, H-1A), 3.00–2.89 (*m*, 3H, H-1*B*,9A,9a), 2.71 (*dd*, *J* = 12.1, 15.6, 1H, H-9B), 2.40 (*dd*, *J* = 6.1, 13.1, 1H, H-3a), 1.10 (*d*, *J* = 6.8, 3H, H-1-*i*-Pr), 1.07 (*d*, *J* = 6.8, 3H, H-3-*i*-Pr) ppm. <sup>13</sup>C {<sup>1</sup>H} NMR (151 MHz, DMSO-*d*<sub>6</sub>, 296 K)  $\delta$  173.0, 172.1, 136.6, 133.2, 130.0, 127.0, 126.1, 46.5, 45.2, 42.4, 41.6, 32.8, 32.0, 19.9, 19.5 ppm. IR (KBr),

$\nu$  (cm<sup>-1</sup>) 3000(OH), 1725 (C=O), 1644 (NCO). Analysis calculate. for C<sub>16</sub>H<sub>19</sub>NO<sub>3</sub>: C, 70.31; H, 7.01; N, 5.12. Found: C, 70.35; H, 6.93; N, 5.08.

Synthesis of (III): *N*-[(2*E*)-3-phenylprop-2-en-1-yl]aniline (0.28 g, 1.28 mmol) was dissolved in 1,4-dioxane (15 ml), and bromomaleic anhydride (0.25 g, 1.41 mmol) was added. The reaction mixture was refluxed for 8 h. After cooling to room temperature, the solvent was removed under reduced pressure. Ethanol (3 ml) was then added to the residue, resulting in the formation of a precipitate of product, which was collected by filtration, washed with ethanol (2 × 1 ml), and air-dried to afford the target compound as a white solid (0.12 g, 0.38 mmol, 30%, m.p. 513–514 K). A single crystal of (III) suitable for X-ray diffraction analysis was grown from an EtOH/DMF solution.

<sup>1</sup>H NMR (600 MHz, DMSO-*d*<sub>6</sub>, 294 K) (*J*, Hz):  $\delta$  12.72 (*s*, 1H, COOH), 7.80 (*dd*, *J* = 1.0, 8.6, 2H, H-2,6-Ph), 7.53 (*dd*, *J* = 1.5, 7.1, 1H, H-5), 7.41–7.28 (*m*, 5H, H-6,7,8, H-3,5-Ph), 7.11 (*dt*, *J* = 1.0, 7.1, 1H, H-4-Ph), 4.75 (*d*, *J* = 18.7, 1H, H-1A), 4.61 (*d*, *J* = 2.0, 18.7, 1H, H-1B), 4.55–4.53 (*m*, 1H, H-4), 3.75 (*dd*, *J* = 4.0, 22.5, 1H, H-9A), 3.74 (*dd*, *J* = 4.0, 22.5, 1H, H-9B) ppm. <sup>13</sup>C {<sup>1</sup>H} NMR (151 MHz, DMSO-*d*<sub>6</sub>, 296 K)  $\delta$  172.1, 168.3, 150.6, 139.5, 132.2, 131.1, 129.3, 129.0 (2C), 128.5, 128.4, 127.5, 126.8, 123.3, 118.0 (2C), 52.7, 42.6, 28.8 ppm. IR (KBr),  $\nu$  (cm<sup>-1</sup>) 3042 (COOH), 1739 (C=O). Analysis calculated for C<sub>19</sub>H<sub>15</sub>NO<sub>3</sub>: C, 74.74; H, 4.95; N, 4.59. Found: C, 74.68; H, 5.03; N, 4.65.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. All hydrogen atoms bound to carbon were geometrically placed and refined using a riding model [ $C-H = 0.95-1.00$  Å,  $U_{iso}(H) = 1.2$  or  $1.5 U_{eq}(C)$ ]. For (I) and (II), the hydrogen atoms of the OH groups were found from difference-Fourier maps and refined as riding atoms in their as-found relative positions with  $U_{iso}(H) = 1.5 U_{eq}(O)$ . For (III), the position of the hydrogen atom of the OH group was calculated geometrically (AFIX 147 card in *SHELXL*) and refined using a riding model with  $U_{iso}(H) = 1.5 U_{eq}(O)$ . Some reflections blocked by the beam stop were omitted [ten reflections for (I), five reflections for (II) and four reflections for (III)].

## Acknowledgements

The authors' contributions are as follows; conceptualization AVG, MA and GMM; synthesis, ENT and ASG; X-ray analysis AVG and KAA; founding KAA; writing (review and editing of the manuscript) KAA and MA; supervision AVG, MA and GMM.

## Funding information

This publication has been supported by the RUDN University Scientific Projects Grant System, project No. 021408-2-000.

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## supporting information

*Acta Cryst.* (2026). E82, 40-46 [https://doi.org/10.1107/S2056989025010709]

## Consistent supramolecular motif of C(7) O—H···O hydrogen-bonded chains and different local symmetries in three isoindole-4-carboxylic acid derivatives

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### Computing details

(3aRS,4RS,9aSR)-3-Oxo-2-(2-phenylethyl)-2,3,3a,4,9,9a-hexahydro-1H-benzo[*f*]isoindole-4-carboxylic acid (I)

#### Crystal data

C<sub>21</sub>H<sub>21</sub>NO<sub>3</sub>  
M<sub>r</sub> = 335.39

Monoclinic, *P*2<sub>1</sub>/*n*  
*a* = 17.3659 (15) Å  
*b* = 8.0220 (7) Å  
*c* = 25.883 (2) Å  
*β* = 109.250 (4)°  
*V* = 3404.2 (5) Å<sup>3</sup>  
*Z* = 8

*F*(000) = 1424  
*D*<sub>x</sub> = 1.309 Mg m<sup>-3</sup>  
Mo *Kα* radiation, λ = 0.71073 Å  
Cell parameters from 3824 reflections  
*θ* = 3.1–20.6°  
μ = 0.09 mm<sup>-1</sup>  
*T* = 150 K  
Block, colourless  
0.26 × 0.24 × 0.21 mm

#### Data collection

Bruker APEXII CCD  
diffractometer  
φ and ω scans  
Absorption correction: multi-scan  
(SADABS; Krause *et al.*, 2015)  
*T*<sub>min</sub> = 0.964, *T*<sub>max</sub> = 0.973  
46007 measured reflections

6513 independent reflections  
3839 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.115  
*θ*<sub>max</sub> = 25.8°, *θ*<sub>min</sub> = 2.5°  
*h* = -21→20  
*k* = -8→9  
*l* = -31→31

#### Refinement

Refinement on *F*<sup>2</sup>  
Least-squares matrix: full  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.048  
*wR*(*F*<sup>2</sup>) = 0.118  
*S* = 1.01  
6513 reflections  
453 parameters  
0 restraints  
Primary atom site location: difference Fourier  
map  
Secondary atom site location: difference Fourier  
map

Hydrogen site location: mixed  
H-atom parameters constrained  
*w* = 1/[σ<sup>2</sup>(*F*<sub>o</sub><sup>2</sup>) + (0.0479*P*)<sup>2</sup> + 0.106*P*]  
where *P* = (*F*<sub>o</sub><sup>2</sup> + 2*F*<sub>c</sub><sup>2</sup>)/3  
(Δ/σ)<sub>max</sub> < 0.001  
Δρ<sub>max</sub> = 0.25 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -0.24 e Å<sup>-3</sup>  
Extinction correction: SHELXL-2019/2  
(Sheldrick, 2008),  
*F*<sub>c</sub>\* = *kF*<sub>c</sub>[1 + 0.001 × *F*<sub>c</sub><sup>2</sup>λ<sup>3</sup>/sin(2θ)]<sup>-1/4</sup>  
Extinction coefficient: 0.0022 (4)

*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 ( $\text{\AA}^2$ )*

|      | <i>x</i>      | <i>y</i>   | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|---------------|------------|--------------|----------------------------------|
| C1   | 0.00531 (13)  | 0.8263 (3) | 0.37497 (9)  | 0.0267 (6)                       |
| H1A  | -0.027011     | 0.727334   | 0.357639     | 0.032*                           |
| H1B  | -0.025994     | 0.928424   | 0.359831     | 0.032*                           |
| C2   | 0.02821 (13)  | 0.8200 (3) | 0.43681 (9)  | 0.0225 (5)                       |
| H2   | 0.040733      | 0.936136   | 0.451174     | 0.027*                           |
| C3   | -0.02924 (14) | 0.7432 (3) | 0.46274 (10) | 0.0256 (6)                       |
| H3A  | -0.048201     | 0.633629   | 0.445696     | 0.031*                           |
| H3B  | -0.077502     | 0.815969   | 0.456545     | 0.031*                           |
| C4   | 0.01362 (13)  | 0.7211 (3) | 0.52363 (10) | 0.0228 (5)                       |
| C5   | -0.03361 (14) | 0.7005 (2) | 0.55757 (10) | 0.0243 (6)                       |
| H5   | -0.091370     | 0.702577   | 0.541975     | 0.029*                           |
| C6   | 0.00147 (15)  | 0.6773 (3) | 0.61309 (10) | 0.0302 (6)                       |
| H6   | -0.032011     | 0.660899   | 0.635202     | 0.036*                           |
| C7   | 0.08581 (15)  | 0.6778 (3) | 0.63680 (10) | 0.0299 (6)                       |
| H7   | 0.110444      | 0.663129   | 0.675156     | 0.036*                           |
| C8   | 0.13333 (15)  | 0.6999 (3) | 0.60377 (10) | 0.0267 (6)                       |
| H8   | 0.191000      | 0.702338   | 0.619989     | 0.032*                           |
| C9   | 0.09887 (14)  | 0.7186 (3) | 0.54735 (9)  | 0.0212 (5)                       |
| C10  | 0.15700 (13)  | 0.7315 (3) | 0.51393 (9)  | 0.0207 (5)                       |
| H10  | 0.193934      | 0.632332   | 0.523065     | 0.025*                           |
| C11  | 0.10774 (13)  | 0.7221 (3) | 0.45326 (9)  | 0.0199 (5)                       |
| H11  | 0.092238      | 0.602434   | 0.445372     | 0.024*                           |
| C12  | 0.14558 (14)  | 0.7756 (3) | 0.41154 (10) | 0.0228 (5)                       |
| C13  | 0.20944 (14)  | 0.8854 (3) | 0.53104 (10) | 0.0224 (5)                       |
| C14  | 0.09812 (15)  | 0.8727 (3) | 0.31641 (9)  | 0.0272 (6)                       |
| H14A | 0.051839      | 0.831791   | 0.285140     | 0.033*                           |
| H14B | 0.148479      | 0.818717   | 0.314697     | 0.033*                           |
| C15  | 0.10546 (16)  | 1.0592 (3) | 0.31188 (10) | 0.0319 (6)                       |
| H15A | 0.145507      | 1.100805   | 0.346298     | 0.038*                           |
| H15B | 0.052094      | 1.110252   | 0.308441     | 0.038*                           |
| C16  | 0.13121 (13)  | 1.1178 (3) | 0.26455 (9)  | 0.0254 (6)                       |
| C17  | 0.12527 (14)  | 1.0192 (3) | 0.21935 (10) | 0.0308 (6)                       |
| H17  | 0.103962      | 0.909440   | 0.217360     | 0.037*                           |
| C18  | 0.15000 (15)  | 1.0788 (3) | 0.17710 (10) | 0.0358 (7)                       |
| H18  | 0.145687      | 1.009313   | 0.146546     | 0.043*                           |
| C19  | 0.18044 (15)  | 1.2360 (3) | 0.17893 (10) | 0.0368 (7)                       |
| H19  | 0.196701      | 1.276584   | 0.149623     | 0.044*                           |
| C20  | 0.18753 (15)  | 1.3364 (3) | 0.22390 (11) | 0.0367 (7)                       |
| H20  | 0.209018      | 1.445889   | 0.225569     | 0.044*                           |

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|      |              |              |              |             |
|------|--------------|--------------|--------------|-------------|
| C21  | 0.16339 (14) | 1.2772 (3)   | 0.26620 (10) | 0.0324 (6)  |
| H21  | 0.168847     | 1.346437     | 0.296974     | 0.039*      |
| N1   | 0.08530 (11) | 0.8272 (2)   | 0.36734 (8)  | 0.0252 (5)  |
| O1   | 0.21861 (9)  | 0.76834 (18) | 0.41499 (6)  | 0.0271 (4)  |
| O3   | 0.27549 (10) | 0.8855 (2)   | 0.56610 (7)  | 0.0366 (4)  |
| O2   | 0.17441 (9)  | 1.02119 (18) | 0.50437 (6)  | 0.0284 (4)  |
| H2O  | 0.199197     | 1.104154     | 0.522907     | 0.043*      |
| C22  | 0.45186 (13) | 0.3870 (3)   | 0.60586 (9)  | 0.0227 (5)  |
| H22A | 0.480811     | 0.298001     | 0.631388     | 0.027*      |
| H22B | 0.480042     | 0.494575     | 0.617795     | 0.027*      |
| C23  | 0.44539 (13) | 0.3463 (3)   | 0.54738 (9)  | 0.0194 (5)  |
| H23  | 0.433400     | 0.452307     | 0.525853     | 0.023*      |
| C24  | 0.51382 (13) | 0.2595 (3)   | 0.53475 (9)  | 0.0218 (5)  |
| H24A | 0.529278     | 0.156929     | 0.557025     | 0.026*      |
| H24B | 0.562126     | 0.333408     | 0.543784     | 0.026*      |
| C25  | 0.48595 (13) | 0.2158 (2)   | 0.47455 (9)  | 0.0203 (5)  |
| C26  | 0.54492 (14) | 0.1906 (2)   | 0.44950 (10) | 0.0236 (5)  |
| H26  | 0.600898     | 0.203525     | 0.470471     | 0.028*      |
| C27  | 0.52385 (14) | 0.1472 (3)   | 0.39495 (10) | 0.0263 (6)  |
| H27  | 0.565010     | 0.129243     | 0.378825     | 0.032*      |
| C28  | 0.44271 (15) | 0.1301 (3)   | 0.36404 (10) | 0.0291 (6)  |
| H28  | 0.427572     | 0.100903     | 0.326439     | 0.035*      |
| C29  | 0.38336 (14) | 0.1557 (3)   | 0.38823 (10) | 0.0257 (6)  |
| H29  | 0.327542     | 0.143902     | 0.366743     | 0.031*      |
| C30  | 0.40352 (13) | 0.1983 (2)   | 0.44307 (9)  | 0.0201 (5)  |
| C31  | 0.33357 (13) | 0.2208 (3)   | 0.46654 (9)  | 0.0203 (5)  |
| H31  | 0.300024     | 0.116756     | 0.458484     | 0.024*      |
| C32  | 0.36894 (13) | 0.2386 (3)   | 0.52879 (9)  | 0.0192 (5)  |
| H32  | 0.384742     | 0.124478     | 0.544220     | 0.023*      |
| C33  | 0.31636 (14) | 0.3142 (2)   | 0.55860 (9)  | 0.0195 (5)  |
| C34  | 0.27825 (14) | 0.3646 (3)   | 0.43917 (9)  | 0.0210 (5)  |
| C35  | 0.33808 (15) | 0.4828 (3)   | 0.64180 (10) | 0.0282 (6)  |
| H35A | 0.278265     | 0.469746     | 0.631919     | 0.034*      |
| H35B | 0.350019     | 0.603238     | 0.640875     | 0.034*      |
| C36  | 0.37905 (15) | 0.4170 (3)   | 0.69935 (10) | 0.0313 (6)  |
| H36A | 0.438734     | 0.432398     | 0.709271     | 0.038*      |
| H36B | 0.360449     | 0.483667     | 0.725182     | 0.038*      |
| C37  | 0.36169 (16) | 0.2360 (3)   | 0.70593 (10) | 0.0300 (6)  |
| C38  | 0.28263 (17) | 0.1814 (3)   | 0.69700 (11) | 0.0409 (7)  |
| H38  | 0.239231     | 0.259835     | 0.686347     | 0.049*      |
| C39  | 0.2653 (2)   | 0.0168 (4)   | 0.70315 (13) | 0.0569 (9)  |
| H39  | 0.210617     | -0.017631    | 0.696692     | 0.068*      |
| C40  | 0.3272 (3)   | -0.0968 (4)  | 0.71858 (13) | 0.0648 (10) |
| H40  | 0.315728     | -0.210446    | 0.723445     | 0.078*      |
| C41  | 0.4059 (2)   | -0.0468 (4)  | 0.72706 (12) | 0.0616 (10) |
| H41  | 0.448742     | -0.126514    | 0.737272     | 0.074*      |
| C42  | 0.42356 (18) | 0.1196 (3)   | 0.72083 (10) | 0.0434 (7)  |
| H42  | 0.478296     | 0.153178     | 0.726845     | 0.052*      |

|     |              |              |             |            |
|-----|--------------|--------------|-------------|------------|
| N2  | 0.36555 (11) | 0.3963 (2)   | 0.60185 (7) | 0.0213 (4) |
| O4  | 0.24114 (9)  | 0.30285 (17) | 0.54630 (6) | 0.0256 (4) |
| O5  | 0.30897 (9)  | 0.51159 (18) | 0.45799 (6) | 0.0284 (4) |
| H5O | 0.281478     | 0.600091     | 0.447130    | 0.043*     |
| O6  | 0.21283 (10) | 0.3465 (2)   | 0.40449 (7) | 0.0380 (5) |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C1  | 0.0245 (13) | 0.0277 (13) | 0.0275 (14) | -0.0016 (11) | 0.0079 (11) | 0.0041 (11)  |
| C2  | 0.0208 (13) | 0.0213 (13) | 0.0268 (14) | 0.0013 (10)  | 0.0099 (11) | 0.0034 (10)  |
| C3  | 0.0219 (13) | 0.0220 (13) | 0.0346 (15) | 0.0023 (10)  | 0.0116 (12) | 0.0039 (11)  |
| C4  | 0.0245 (13) | 0.0135 (12) | 0.0322 (15) | 0.0003 (10)  | 0.0117 (12) | 0.0037 (10)  |
| C5  | 0.0268 (13) | 0.0139 (12) | 0.0380 (16) | 0.0012 (10)  | 0.0187 (12) | 0.0031 (11)  |
| C6  | 0.0418 (17) | 0.0205 (13) | 0.0406 (17) | -0.0008 (11) | 0.0302 (14) | 0.0003 (11)  |
| C7  | 0.0417 (16) | 0.0291 (14) | 0.0223 (14) | -0.0005 (12) | 0.0151 (13) | -0.0025 (11) |
| C8  | 0.0286 (14) | 0.0265 (13) | 0.0282 (15) | 0.0013 (11)  | 0.0134 (12) | -0.0001 (11) |
| C9  | 0.0266 (13) | 0.0147 (12) | 0.0272 (14) | 0.0007 (10)  | 0.0154 (11) | 0.0010 (10)  |
| C10 | 0.0220 (13) | 0.0186 (12) | 0.0251 (14) | 0.0037 (10)  | 0.0125 (11) | 0.0044 (10)  |
| C11 | 0.0215 (13) | 0.0147 (12) | 0.0256 (14) | -0.0023 (9)  | 0.0108 (11) | 0.0005 (10)  |
| C12 | 0.0308 (15) | 0.0138 (12) | 0.0273 (14) | 0.0039 (10)  | 0.0146 (12) | 0.0021 (10)  |
| C13 | 0.0188 (13) | 0.0267 (14) | 0.0255 (14) | 0.0044 (11)  | 0.0126 (12) | 0.0027 (11)  |
| C14 | 0.0325 (14) | 0.0290 (14) | 0.0207 (14) | 0.0024 (11)  | 0.0096 (12) | 0.0028 (11)  |
| C15 | 0.0443 (16) | 0.0298 (14) | 0.0254 (15) | -0.0031 (12) | 0.0167 (13) | -0.0005 (11) |
| C16 | 0.0240 (13) | 0.0309 (14) | 0.0210 (14) | 0.0013 (11)  | 0.0072 (11) | 0.0023 (11)  |
| C17 | 0.0331 (15) | 0.0332 (15) | 0.0247 (15) | 0.0020 (11)  | 0.0074 (12) | 0.0019 (12)  |
| C18 | 0.0349 (16) | 0.0507 (18) | 0.0221 (15) | 0.0075 (13)  | 0.0100 (13) | 0.0025 (12)  |
| C19 | 0.0247 (14) | 0.0597 (19) | 0.0264 (16) | 0.0015 (13)  | 0.0090 (12) | 0.0148 (14)  |
| C20 | 0.0295 (15) | 0.0429 (17) | 0.0347 (17) | -0.0076 (12) | 0.0066 (13) | 0.0137 (13)  |
| C21 | 0.0316 (15) | 0.0362 (15) | 0.0266 (15) | -0.0045 (12) | 0.0057 (12) | 0.0007 (12)  |
| N1  | 0.0237 (11) | 0.0283 (11) | 0.0248 (11) | 0.0027 (9)   | 0.0096 (9)  | 0.0066 (9)   |
| O1  | 0.0259 (10) | 0.0283 (9)  | 0.0329 (10) | 0.0077 (7)   | 0.0175 (8)  | 0.0077 (7)   |
| O3  | 0.0230 (10) | 0.0379 (10) | 0.0423 (12) | -0.0019 (8)  | 0.0017 (9)  | 0.0059 (9)   |
| O2  | 0.0243 (9)  | 0.0184 (9)  | 0.0390 (11) | -0.0010 (7)  | 0.0057 (8)  | -0.0008 (7)  |
| C22 | 0.0245 (13) | 0.0207 (12) | 0.0223 (13) | -0.0016 (10) | 0.0070 (11) | -0.0011 (10) |
| C23 | 0.0219 (12) | 0.0176 (12) | 0.0194 (13) | -0.0016 (10) | 0.0078 (10) | 0.0003 (10)  |
| C24 | 0.0200 (13) | 0.0207 (12) | 0.0242 (13) | -0.0023 (10) | 0.0068 (11) | 0.0004 (10)  |
| C25 | 0.0238 (13) | 0.0133 (12) | 0.0260 (14) | 0.0002 (9)   | 0.0112 (11) | 0.0013 (10)  |
| C26 | 0.0237 (13) | 0.0169 (12) | 0.0335 (15) | 0.0008 (10)  | 0.0140 (12) | 0.0033 (11)  |
| C27 | 0.0314 (15) | 0.0210 (13) | 0.0338 (15) | 0.0037 (11)  | 0.0205 (13) | 0.0020 (11)  |
| C28 | 0.0408 (16) | 0.0261 (14) | 0.0232 (14) | 0.0040 (12)  | 0.0143 (13) | -0.0017 (11) |
| C29 | 0.0271 (14) | 0.0242 (13) | 0.0252 (14) | 0.0019 (11)  | 0.0079 (11) | -0.0026 (11) |
| C30 | 0.0245 (13) | 0.0126 (11) | 0.0253 (13) | 0.0014 (10)  | 0.0110 (11) | -0.0003 (10) |
| C31 | 0.0214 (12) | 0.0180 (12) | 0.0214 (13) | -0.0017 (10) | 0.0072 (10) | -0.0016 (10) |
| C32 | 0.0227 (13) | 0.0156 (11) | 0.0204 (13) | 0.0000 (9)   | 0.0086 (11) | 0.0006 (9)   |
| C33 | 0.0255 (14) | 0.0119 (11) | 0.0230 (13) | -0.0011 (10) | 0.0102 (11) | 0.0026 (10)  |
| C34 | 0.0192 (13) | 0.0256 (14) | 0.0211 (13) | -0.0002 (10) | 0.0105 (11) | -0.0019 (10) |
| C35 | 0.0371 (15) | 0.0217 (13) | 0.0301 (15) | 0.0014 (11)  | 0.0168 (12) | -0.0037 (11) |

|     |             |             |             |              |             |              |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C36 | 0.0404 (16) | 0.0319 (15) | 0.0250 (15) | -0.0015 (12) | 0.0155 (13) | -0.0055 (11) |
| C37 | 0.0426 (16) | 0.0306 (14) | 0.0217 (14) | 0.0048 (12)  | 0.0171 (12) | -0.0014 (11) |
| C38 | 0.0528 (19) | 0.0344 (16) | 0.0426 (18) | -0.0021 (14) | 0.0251 (15) | -0.0026 (13) |
| C39 | 0.085 (2)   | 0.0398 (19) | 0.062 (2)   | -0.0143 (18) | 0.047 (2)   | -0.0062 (16) |
| C40 | 0.133 (3)   | 0.0323 (18) | 0.055 (2)   | -0.002 (2)   | 0.067 (2)   | 0.0016 (16)  |
| C41 | 0.109 (3)   | 0.043 (2)   | 0.048 (2)   | 0.035 (2)    | 0.047 (2)   | 0.0138 (15)  |
| C42 | 0.0569 (19) | 0.0497 (18) | 0.0289 (16) | 0.0168 (15)  | 0.0214 (14) | 0.0085 (13)  |
| N2  | 0.0226 (11) | 0.0223 (10) | 0.0209 (11) | -0.0001 (8)  | 0.0095 (9)  | -0.0025 (8)  |
| O4  | 0.0196 (9)  | 0.0242 (9)  | 0.0352 (10) | -0.0025 (7)  | 0.0121 (8)  | -0.0029 (7)  |
| O5  | 0.0245 (9)  | 0.0158 (9)  | 0.0382 (11) | 0.0010 (7)   | 0.0015 (8)  | 0.0014 (7)   |
| O6  | 0.0287 (10) | 0.0371 (11) | 0.0366 (11) | 0.0006 (8)   | -0.0050 (9) | -0.0044 (8)  |

*Geometric parameters (Å, °)*

|          |           |          |           |
|----------|-----------|----------|-----------|
| C1—N1    | 1.466 (3) | C22—N2   | 1.470 (3) |
| C1—C2    | 1.518 (3) | C22—C23  | 1.516 (3) |
| C1—H1A   | 0.9900    | C22—H22A | 0.9900    |
| C1—H1B   | 0.9900    | C22—H22B | 0.9900    |
| C2—C3    | 1.505 (3) | C23—C24  | 1.503 (3) |
| C2—C11   | 1.522 (3) | C23—C32  | 1.523 (3) |
| C2—H2    | 1.0000    | C23—H23  | 1.0000    |
| C3—C4    | 1.515 (3) | C24—C25  | 1.513 (3) |
| C3—H3A   | 0.9900    | C24—H24A | 0.9900    |
| C3—H3B   | 0.9900    | C24—H24B | 0.9900    |
| C4—C5    | 1.395 (3) | C25—C26  | 1.396 (3) |
| C4—C9    | 1.404 (3) | C25—C30  | 1.401 (3) |
| C5—C6    | 1.376 (3) | C26—C27  | 1.381 (3) |
| C5—H5    | 0.9500    | C26—H26  | 0.9500    |
| C6—C7    | 1.389 (3) | C27—C28  | 1.379 (3) |
| C6—H6    | 0.9500    | C27—H27  | 0.9500    |
| C7—C8    | 1.381 (3) | C28—C29  | 1.386 (3) |
| C7—H7    | 0.9500    | C28—H28  | 0.9500    |
| C8—C9    | 1.392 (3) | C29—C30  | 1.388 (3) |
| C8—H8    | 0.9500    | C29—H29  | 0.9500    |
| C9—C10   | 1.534 (3) | C30—C31  | 1.538 (3) |
| C10—C13  | 1.511 (3) | C31—C34  | 1.518 (3) |
| C10—C11  | 1.522 (3) | C31—C32  | 1.530 (3) |
| C10—H10  | 1.0000    | C31—H31  | 1.0000    |
| C11—C12  | 1.500 (3) | C32—C33  | 1.504 (3) |
| C11—H11  | 1.0000    | C32—H32  | 1.0000    |
| C12—O1   | 1.243 (3) | C33—O4   | 1.242 (2) |
| C12—N1   | 1.336 (3) | C33—N2   | 1.336 (3) |
| C13—O3   | 1.205 (3) | C34—O6   | 1.202 (3) |
| C13—O2   | 1.325 (3) | C34—O5   | 1.320 (3) |
| C14—N1   | 1.454 (3) | C35—N2   | 1.450 (3) |
| C14—C15  | 1.509 (3) | C35—C36  | 1.518 (3) |
| C14—H14A | 0.9900    | C35—H35A | 0.9900    |
| C14—H14B | 0.9900    | C35—H35B | 0.9900    |

|            |             |               |             |
|------------|-------------|---------------|-------------|
| C15—C16    | 1.511 (3)   | C36—C37       | 1.504 (3)   |
| C15—H15A   | 0.9900      | C36—H36A      | 0.9900      |
| C15—H15B   | 0.9900      | C36—H36B      | 0.9900      |
| C16—C17    | 1.388 (3)   | C37—C42       | 1.379 (3)   |
| C16—C21    | 1.390 (3)   | C37—C38       | 1.386 (3)   |
| C17—C18    | 1.385 (3)   | C38—C39       | 1.375 (4)   |
| C17—H17    | 0.9500      | C38—H38       | 0.9500      |
| C18—C19    | 1.362 (4)   | C39—C40       | 1.364 (4)   |
| C18—H18    | 0.9500      | C39—H39       | 0.9500      |
| C19—C20    | 1.387 (4)   | C40—C41       | 1.371 (5)   |
| C19—H19    | 0.9500      | C40—H40       | 0.9500      |
| C20—C21    | 1.379 (3)   | C41—C42       | 1.391 (4)   |
| C20—H20    | 0.9500      | C41—H41       | 0.9500      |
| C21—H21    | 0.9500      | C42—H42       | 0.9500      |
| O2—H2O     | 0.8498      | O5—H5O        | 0.8500      |
|            |             |               |             |
| N1—C1—C2   | 102.23 (17) | N2—C22—C23    | 101.68 (17) |
| N1—C1—H1A  | 111.3       | N2—C22—H22A   | 111.4       |
| C2—C1—H1A  | 111.3       | C23—C22—H22A  | 111.4       |
| N1—C1—H1B  | 111.3       | N2—C22—H22B   | 111.4       |
| C2—C1—H1B  | 111.3       | C23—C22—H22B  | 111.4       |
| H1A—C1—H1B | 109.2       | H22A—C22—H22B | 109.3       |
| C3—C2—C1   | 120.13 (19) | C24—C23—C22   | 120.88 (19) |
| C3—C2—C11  | 109.75 (18) | C24—C23—C32   | 109.73 (17) |
| C1—C2—C11  | 102.13 (17) | C22—C23—C32   | 101.97 (17) |
| C3—C2—H2   | 108.1       | C24—C23—H23   | 107.9       |
| C1—C2—H2   | 108.1       | C22—C23—H23   | 107.9       |
| C11—C2—H2  | 108.1       | C32—C23—H23   | 107.9       |
| C2—C3—C4   | 109.94 (19) | C23—C24—C25   | 108.91 (18) |
| C2—C3—H3A  | 109.7       | C23—C24—H24A  | 109.9       |
| C4—C3—H3A  | 109.7       | C25—C24—H24A  | 109.9       |
| C2—C3—H3B  | 109.7       | C23—C24—H24B  | 109.9       |
| C4—C3—H3B  | 109.7       | C25—C24—H24B  | 109.9       |
| H3A—C3—H3B | 108.2       | H24A—C24—H24B | 108.3       |
| C5—C4—C9   | 118.5 (2)   | C26—C25—C30   | 118.7 (2)   |
| C5—C4—C3   | 118.7 (2)   | C26—C25—C24   | 118.5 (2)   |
| C9—C4—C3   | 122.8 (2)   | C30—C25—C24   | 122.73 (19) |
| C6—C5—C4   | 121.6 (2)   | C27—C26—C25   | 121.6 (2)   |
| C6—C5—H5   | 119.2       | C27—C26—H26   | 119.2       |
| C4—C5—H5   | 119.2       | C25—C26—H26   | 119.2       |
| C5—C6—C7   | 120.0 (2)   | C28—C27—C26   | 119.6 (2)   |
| C5—C6—H6   | 120.0       | C28—C27—H27   | 120.2       |
| C7—C6—H6   | 120.0       | C26—C27—H27   | 120.2       |
| C8—C7—C6   | 119.0 (2)   | C27—C28—C29   | 119.5 (2)   |
| C8—C7—H7   | 120.5       | C27—C28—H28   | 120.2       |
| C6—C7—H7   | 120.5       | C29—C28—H28   | 120.2       |
| C7—C8—C9   | 121.7 (2)   | C28—C29—C30   | 121.6 (2)   |
| C7—C8—H8   | 119.2       | C28—C29—H29   | 119.2       |

|               |             |               |             |
|---------------|-------------|---------------|-------------|
| C9—C8—H8      | 119.2       | C30—C29—H29   | 119.2       |
| C8—C9—C4      | 119.2 (2)   | C29—C30—C25   | 119.0 (2)   |
| C8—C9—C10     | 117.6 (2)   | C29—C30—C31   | 117.9 (2)   |
| C4—C9—C10     | 123.2 (2)   | C25—C30—C31   | 123.2 (2)   |
| C13—C10—C11   | 114.95 (18) | C34—C31—C32   | 113.18 (17) |
| C13—C10—C9    | 109.55 (17) | C34—C31—C30   | 111.44 (17) |
| C11—C10—C9    | 109.14 (18) | C32—C31—C30   | 109.38 (18) |
| C13—C10—H10   | 107.6       | C34—C31—H31   | 107.5       |
| C11—C10—H10   | 107.6       | C32—C31—H31   | 107.5       |
| C9—C10—H10    | 107.6       | C30—C31—H31   | 107.5       |
| C12—C11—C2    | 102.93 (18) | C33—C32—C23   | 102.84 (17) |
| C12—C11—C10   | 119.87 (19) | C33—C32—C31   | 118.48 (18) |
| C2—C11—C10    | 114.36 (18) | C23—C32—C31   | 113.01 (17) |
| C12—C11—H11   | 106.2       | C33—C32—H32   | 107.3       |
| C2—C11—H11    | 106.2       | C23—C32—H32   | 107.3       |
| C10—C11—H11   | 106.2       | C31—C32—H32   | 107.3       |
| O1—C12—N1     | 124.6 (2)   | O4—C33—N2     | 125.4 (2)   |
| O1—C12—C11    | 127.9 (2)   | O4—C33—C32    | 127.2 (2)   |
| N1—C12—C11    | 107.42 (19) | N2—C33—C32    | 107.46 (18) |
| O3—C13—O2     | 123.5 (2)   | O6—C34—O5     | 123.4 (2)   |
| O3—C13—C10    | 123.6 (2)   | O6—C34—C31    | 123.6 (2)   |
| O2—C13—C10    | 113.0 (2)   | O5—C34—C31    | 112.94 (19) |
| N1—C14—C15    | 111.21 (18) | N2—C35—C36    | 112.03 (19) |
| N1—C14—H14A   | 109.4       | N2—C35—H35A   | 109.2       |
| C15—C14—H14A  | 109.4       | C36—C35—H35A  | 109.2       |
| N1—C14—H14B   | 109.4       | N2—C35—H35B   | 109.2       |
| C15—C14—H14B  | 109.4       | C36—C35—H35B  | 109.2       |
| H14A—C14—H14B | 108.0       | H35A—C35—H35B | 107.9       |
| C14—C15—C16   | 115.26 (19) | C37—C36—C35   | 113.6 (2)   |
| C14—C15—H15A  | 108.5       | C37—C36—H36A  | 108.8       |
| C16—C15—H15A  | 108.5       | C35—C36—H36A  | 108.8       |
| C14—C15—H15B  | 108.5       | C37—C36—H36B  | 108.8       |
| C16—C15—H15B  | 108.5       | C35—C36—H36B  | 108.8       |
| H15A—C15—H15B | 107.5       | H36A—C36—H36B | 107.7       |
| C17—C16—C21   | 117.9 (2)   | C42—C37—C38   | 118.1 (2)   |
| C17—C16—C15   | 123.1 (2)   | C42—C37—C36   | 121.2 (2)   |
| C21—C16—C15   | 119.0 (2)   | C38—C37—C36   | 120.7 (2)   |
| C18—C17—C16   | 120.8 (2)   | C39—C38—C37   | 121.7 (3)   |
| C18—C17—H17   | 119.6       | C39—C38—H38   | 119.1       |
| C16—C17—H17   | 119.6       | C37—C38—H38   | 119.1       |
| C19—C18—C17   | 120.6 (2)   | C40—C39—C38   | 119.7 (3)   |
| C19—C18—H18   | 119.7       | C40—C39—H39   | 120.2       |
| C17—C18—H18   | 119.7       | C38—C39—H39   | 120.2       |
| C18—C19—C20   | 119.5 (2)   | C39—C40—C41   | 119.9 (3)   |
| C18—C19—H19   | 120.2       | C39—C40—H40   | 120.1       |
| C20—C19—H19   | 120.2       | C41—C40—H40   | 120.1       |
| C21—C20—C19   | 120.0 (2)   | C40—C41—C42   | 120.6 (3)   |
| C21—C20—H20   | 120.0       | C40—C41—H41   | 119.7       |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C19—C20—H20     | 120.0        | C42—C41—H41     | 119.7        |
| C20—C21—C16     | 121.1 (2)    | C37—C42—C41     | 120.0 (3)    |
| C20—C21—H21     | 119.4        | C37—C42—H42     | 120.0        |
| C16—C21—H21     | 119.4        | C41—C42—H42     | 120.0        |
| C12—N1—C14      | 122.89 (19)  | C33—N2—C35      | 124.28 (19)  |
| C12—N1—C1       | 113.12 (18)  | C33—N2—C22      | 112.91 (17)  |
| C14—N1—C1       | 123.94 (18)  | C35—N2—C22      | 122.79 (18)  |
| C13—O2—H2O      | 106.8        | C34—O5—H5O      | 120.7        |
|                 |              |                 |              |
| N1—C1—C2—C3     | -152.87 (19) | N2—C22—C23—C24  | -155.55 (18) |
| N1—C1—C2—C11    | -31.3 (2)    | N2—C22—C23—C32  | -33.6 (2)    |
| C1—C2—C3—C4     | 168.90 (19)  | C22—C23—C24—C25 | 172.71 (18)  |
| C11—C2—C3—C4    | 51.1 (2)     | C32—C23—C24—C25 | 54.6 (2)     |
| C2—C3—C4—C5     | 160.80 (19)  | C23—C24—C25—C26 | 157.38 (18)  |
| C2—C3—C4—C9     | -19.9 (3)    | C23—C24—C25—C30 | -23.2 (3)    |
| C9—C4—C5—C6     | -0.3 (3)     | C30—C25—C26—C27 | -0.7 (3)     |
| C3—C4—C5—C6     | 179.0 (2)    | C24—C25—C26—C27 | 178.7 (2)    |
| C4—C5—C6—C7     | 1.5 (3)      | C25—C26—C27—C28 | 0.8 (3)      |
| C5—C6—C7—C8     | -0.7 (3)     | C26—C27—C28—C29 | -0.4 (3)     |
| C6—C7—C8—C9     | -1.2 (3)     | C27—C28—C29—C30 | -0.1 (3)     |
| C7—C8—C9—C4     | 2.3 (3)      | C28—C29—C30—C25 | 0.1 (3)      |
| C7—C8—C9—C10    | -176.3 (2)   | C28—C29—C30—C31 | -178.80 (19) |
| C5—C4—C9—C8     | -1.5 (3)     | C26—C25—C30—C29 | 0.3 (3)      |
| C3—C4—C9—C8     | 179.2 (2)    | C24—C25—C30—C29 | -179.13 (19) |
| C5—C4—C9—C10    | 176.96 (19)  | C26—C25—C30—C31 | 179.13 (18)  |
| C3—C4—C9—C10    | -2.3 (3)     | C24—C25—C30—C31 | -0.3 (3)     |
| C8—C9—C10—C13   | -62.7 (2)    | C29—C30—C31—C34 | -63.4 (2)    |
| C4—C9—C10—C13   | 118.8 (2)    | C25—C30—C31—C34 | 117.8 (2)    |
| C8—C9—C10—C11   | 170.67 (18)  | C29—C30—C31—C32 | 170.71 (18)  |
| C4—C9—C10—C11   | -7.9 (3)     | C25—C30—C31—C32 | -8.1 (3)     |
| C3—C2—C11—C12   | 162.37 (18)  | C24—C23—C32—C33 | 163.64 (17)  |
| C1—C2—C11—C12   | 33.9 (2)     | C22—C23—C32—C33 | 34.3 (2)     |
| C3—C2—C11—C10   | -66.0 (2)    | C24—C23—C32—C31 | -67.5 (2)    |
| C1—C2—C11—C10   | 165.54 (18)  | C22—C23—C32—C31 | 163.25 (17)  |
| C13—C10—C11—C12 | 40.6 (3)     | C34—C31—C32—C33 | 36.2 (3)     |
| C9—C10—C11—C12  | 164.08 (18)  | C30—C31—C32—C33 | 161.06 (17)  |
| C13—C10—C11—C2  | -82.3 (2)    | C34—C31—C32—C23 | -84.2 (2)    |
| C9—C10—C11—C2   | 41.2 (2)     | C30—C31—C32—C23 | 40.7 (2)     |
| C2—C11—C12—O1   | 158.7 (2)    | C23—C32—C33—O4  | 158.3 (2)    |
| C10—C11—C12—O1  | 30.4 (3)     | C31—C32—C33—O4  | 32.9 (3)     |
| C2—C11—C12—N1   | -24.2 (2)    | C23—C32—C33—N2  | -22.3 (2)    |
| C10—C11—C12—N1  | -152.48 (19) | C31—C32—C33—N2  | -147.68 (19) |
| C11—C10—C13—O3  | -144.5 (2)   | C32—C31—C34—O6  | -134.0 (2)   |
| C9—C10—C13—O3   | 92.2 (3)     | C30—C31—C34—O6  | 102.2 (2)    |
| C11—C10—C13—O2  | 37.0 (3)     | C32—C31—C34—O5  | 45.0 (2)     |
| C9—C10—C13—O2   | -86.3 (2)    | C30—C31—C34—O5  | -78.7 (2)    |
| N1—C14—C15—C16  | 171.36 (19)  | N2—C35—C36—C37  | -61.4 (3)    |
| C14—C15—C16—C17 | 18.7 (3)     | C35—C36—C37—C42 | 118.4 (3)    |

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C14—C15—C16—C21 | -160.5 (2)   | C35—C36—C37—C38 | -61.5 (3)    |
| C21—C16—C17—C18 | -0.6 (4)     | C42—C37—C38—C39 | 0.6 (4)      |
| C15—C16—C17—C18 | -179.8 (2)   | C36—C37—C38—C39 | -179.5 (2)   |
| C16—C17—C18—C19 | -0.3 (4)     | C37—C38—C39—C40 | 0.2 (4)      |
| C17—C18—C19—C20 | 0.7 (4)      | C38—C39—C40—C41 | -1.0 (5)     |
| C18—C19—C20—C21 | -0.4 (4)     | C39—C40—C41—C42 | 0.9 (5)      |
| C19—C20—C21—C16 | -0.5 (4)     | C38—C37—C42—C41 | -0.7 (4)     |
| C17—C16—C21—C20 | 1.0 (4)      | C36—C37—C42—C41 | 179.5 (2)    |
| C15—C16—C21—C20 | -179.8 (2)   | C40—C41—C42—C37 | -0.1 (4)     |
| O1—C12—N1—C14   | 3.5 (3)      | O4—C33—N2—C35   | 1.1 (3)      |
| C11—C12—N1—C14  | -173.70 (18) | C32—C33—N2—C35  | -178.29 (18) |
| O1—C12—N1—C1    | -178.8 (2)   | O4—C33—N2—C22   | 179.74 (19)  |
| C11—C12—N1—C1   | 4.0 (2)      | C32—C33—N2—C22  | 0.3 (2)      |
| C15—C14—N1—C12  | -98.4 (2)    | C36—C35—N2—C33  | 121.7 (2)    |
| C15—C14—N1—C1   | 84.2 (3)     | C36—C35—N2—C22  | -56.8 (3)    |
| C2—C1—N1—C12    | 17.9 (2)     | C23—C22—N2—C33  | 21.8 (2)     |
| C2—C1—N1—C14    | -164.43 (19) | C23—C22—N2—C35  | -159.60 (18) |

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

Cg3, Cg4, Cg10 and Cg11 are the centroids of the C4—C9, C16—C21, C25—C30 and C37—C42 rings, respectively.

| $D-H\cdots A$                       | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------------------|-------|-------------|-------------|---------------|
| O2—H2O $\cdots$ O4 <sup>i</sup>     | 0.85  | 1.77        | 2.607 (2)   | 166           |
| O5—H5O $\cdots$ O1                  | 0.85  | 1.76        | 2.606 (2)   | 171           |
| C2—H2 $\cdots$ O2                   | 1.00  | 2.38        | 3.030 (3)   | 122           |
| C15—H15A $\cdots$ O6 <sup>i</sup>   | 0.99  | 2.52        | 3.404 (3)   | 148           |
| C23—H23 $\cdots$ O5                 | 1.00  | 2.34        | 3.018 (3)   | 124           |
| C3—H3A $\cdots$ Cg3 <sup>ii</sup>   | 0.99  | 2.75        | 3.702 (3)   | 161           |
| C7—H7 $\cdots$ Cg11 <sup>iii</sup>  | 0.95  | 2.86        | 3.796 (3)   | 167           |
| C20—H20 $\cdots$ Cg4 <sup>iv</sup>  | 0.95  | 2.95        | 3.783 (3)   | 148           |
| C24—H24A $\cdots$ Cg10 <sup>v</sup> | 0.99  | 2.71        | 3.648 (3)   | 158           |

Symmetry codes: (i)  $x, y+1, z$ ; (ii)  $-x, -y+1, -z+1$ ; (iii)  $-x+1/2, y+1/2, -z+3/2$ ; (iv)  $-x+1/2, y+1/2, -z+1/2$ ; (v)  $-x+1, -y, -z+1$ .

### (3aRS,4RS,9aSR)-3-Oxo-2-(propan-2-yl)-2,3,3a,4,9,9a-hexahydro-1H-benzo[*f*]isoindole-4-carboxylic acid (II)

#### Crystal data

$C_{16}H_{19}NO_3$

$M_r = 273.32$

Monoclinic,  $C2/c$

$a = 25.1799$  (12)  $\text{\AA}$

$b = 7.9085$  (3)  $\text{\AA}$

$c = 17.6079$  (8)  $\text{\AA}$

$\beta = 126.771$  (2) $^\circ$

$V = 2808.7$  (2)  $\text{\AA}^3$

$Z = 8$

$F(000) = 1168$

$D_x = 1.293$   $\text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$   $\text{\AA}$

Cell parameters from 2994 reflections

$\theta = 2.8\text{--}25.0^\circ$

$\mu = 0.09$   $\text{mm}^{-1}$

$T = 150$  K

Block, colourless

$0.31 \times 0.29 \times 0.29$  mm

*Data collection*

Bruker APEXII CCD  
diffractometer

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.964$ ,  $T_{\max} = 0.967$

12572 measured reflections

3240 independent reflections

2416 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 3.3^\circ$

$h = -32 \rightarrow 31$

$k = -10 \rightarrow 10$

$l = -22 \rightarrow 22$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.108$

$S = 1.02$

3240 reflections

184 parameters

0 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier  
map

Hydrogen site location: mixed

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 1.5162P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.32 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$

Extinction correction: SHELXL-2019/2  
(Sheldrick, 2008),

$F_c^* = kFc[1 + 0.001x\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0014 (4)

*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 ( $\text{\AA}^2$ )*

|     | <i>x</i>    | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|--------------|--------------|----------------------------------|
| C1  | 0.60850 (7) | 0.61571 (19) | 0.29553 (10) | 0.0232 (3)                       |
| H1A | 0.583765    | 0.518919     | 0.295989     | 0.028*                           |
| H1B | 0.592352    | 0.721854     | 0.305077     | 0.028*                           |
| C2  | 0.68282 (7) | 0.59480 (17) | 0.36994 (9)  | 0.0192 (3)                       |
| H2  | 0.704221    | 0.708128     | 0.382438     | 0.023*                           |
| C3  | 0.70846 (7) | 0.51448 (18) | 0.46394 (10) | 0.0224 (3)                       |
| H3A | 0.682432    | 0.411449     | 0.453086     | 0.027*                           |
| H3B | 0.703078    | 0.594238     | 0.502227     | 0.027*                           |
| C4  | 0.78093 (7) | 0.46863 (17) | 0.51731 (10) | 0.0215 (3)                       |
| C5  | 0.81858 (8) | 0.44082 (18) | 0.61502 (10) | 0.0258 (3)                       |
| H5  | 0.798500    | 0.456195     | 0.646134     | 0.031*                           |
| C6  | 0.88425 (8) | 0.39156 (19) | 0.66755 (11) | 0.0291 (4)                       |
| H6  | 0.908868    | 0.374215     | 0.733961     | 0.035*                           |
| C7  | 0.91399 (8) | 0.36760 (19) | 0.62300 (11) | 0.0303 (4)                       |
| H7  | 0.958918    | 0.332208     | 0.658469     | 0.036*                           |
| C8  | 0.87778 (7) | 0.39557 (19) | 0.52631 (11) | 0.0267 (3)                       |
| H8  | 0.898407    | 0.379403     | 0.496007     | 0.032*                           |
| C9  | 0.81166 (7) | 0.44700 (17) | 0.47256 (10) | 0.0208 (3)                       |
| C10 | 0.77554 (7) | 0.47589 (18) | 0.36596 (10) | 0.0199 (3)                       |
| H10 | 0.783543    | 0.374453     | 0.340160     | 0.024*                           |

|      |             |              |              |            |
|------|-------------|--------------|--------------|------------|
| C11  | 0.70147 (7) | 0.48662 (17) | 0.31745 (9)  | 0.0184 (3) |
| H11  | 0.686573    | 0.369028     | 0.316722     | 0.022*     |
| C12  | 0.65519 (7) | 0.55150 (17) | 0.21746 (10) | 0.0198 (3) |
| C13  | 0.80492 (7) | 0.62868 (18) | 0.35041 (10) | 0.0227 (3) |
| C14  | 0.54115 (7) | 0.6643 (2)   | 0.11489 (10) | 0.0276 (3) |
| H10A | 0.548602    | 0.645306     | 0.065779     | 0.033*     |
| C15  | 0.48583 (9) | 0.5474 (3)   | 0.09262 (13) | 0.0466 (5) |
| H15A | 0.499459    | 0.429709     | 0.096702     | 0.070*     |
| H15B | 0.445963    | 0.570504     | 0.028408     | 0.070*     |
| H15C | 0.476272    | 0.566514     | 0.138330     | 0.070*     |
| C16  | 0.52363 (9) | 0.8486 (2)   | 0.11078 (13) | 0.0441 (5) |
| H16A | 0.561092    | 0.919211     | 0.127077     | 0.066*     |
| H16B | 0.513755    | 0.869581     | 0.156005     | 0.066*     |
| H16C | 0.484672    | 0.876346     | 0.046602     | 0.066*     |
| N1   | 0.60273 (6) | 0.62084 (15) | 0.20737 (8)  | 0.0225 (3) |
| O1   | 0.66190 (5) | 0.53845 (13) | 0.15330 (7)  | 0.0242 (3) |
| O2   | 0.78009 (5) | 0.77364 (13) | 0.35226 (8)  | 0.0288 (3) |
| H2O  | 0.802114    | 0.854226     | 0.350604     | 0.043*     |
| O3   | 0.84684 (7) | 0.61777 (16) | 0.33814 (11) | 0.0513 (4) |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$    | $U^{13}$   | $U^{23}$    |
|-----|-------------|-------------|-------------|-------------|------------|-------------|
| C1  | 0.0273 (8)  | 0.0241 (8)  | 0.0252 (7)  | 0.0013 (6)  | 0.0195 (7) | -0.0003 (6) |
| C2  | 0.0241 (7)  | 0.0168 (7)  | 0.0218 (7)  | -0.0006 (6) | 0.0165 (6) | -0.0019 (6) |
| C3  | 0.0289 (8)  | 0.0214 (7)  | 0.0224 (7)  | -0.0019 (6) | 0.0183 (7) | -0.0017 (6) |
| C4  | 0.0299 (8)  | 0.0133 (7)  | 0.0224 (7)  | -0.0037 (6) | 0.0163 (6) | -0.0018 (6) |
| C5  | 0.0375 (9)  | 0.0177 (7)  | 0.0235 (7)  | -0.0047 (6) | 0.0191 (7) | -0.0026 (6) |
| C6  | 0.0360 (9)  | 0.0207 (8)  | 0.0194 (7)  | -0.0068 (7) | 0.0106 (7) | -0.0003 (6) |
| C7  | 0.0238 (8)  | 0.0277 (8)  | 0.0281 (8)  | -0.0042 (6) | 0.0094 (7) | 0.0031 (7)  |
| C8  | 0.0262 (8)  | 0.0246 (8)  | 0.0281 (8)  | -0.0030 (6) | 0.0156 (7) | 0.0015 (6)  |
| C9  | 0.0258 (8)  | 0.0146 (7)  | 0.0216 (7)  | -0.0032 (6) | 0.0140 (6) | -0.0006 (6) |
| C10 | 0.0240 (7)  | 0.0181 (7)  | 0.0208 (7)  | 0.0017 (6)  | 0.0150 (6) | 0.0004 (6)  |
| C11 | 0.0227 (7)  | 0.0156 (6)  | 0.0203 (7)  | -0.0005 (5) | 0.0147 (6) | -0.0006 (5) |
| C12 | 0.0237 (7)  | 0.0149 (7)  | 0.0224 (7)  | 0.0003 (5)  | 0.0148 (6) | -0.0013 (6) |
| C13 | 0.0243 (7)  | 0.0265 (8)  | 0.0216 (7)  | 0.0017 (6)  | 0.0161 (6) | 0.0025 (6)  |
| C14 | 0.0235 (8)  | 0.0359 (9)  | 0.0222 (7)  | 0.0048 (6)  | 0.0130 (7) | -0.0013 (6) |
| C15 | 0.0351 (10) | 0.0659 (13) | 0.0332 (9)  | -0.0163 (9) | 0.0173 (8) | -0.0145 (9) |
| C16 | 0.0356 (10) | 0.0421 (11) | 0.0364 (10) | 0.0152 (8)  | 0.0119 (8) | 0.0049 (8)  |
| N1  | 0.0240 (7)  | 0.0254 (6)  | 0.0212 (6)  | 0.0041 (5)  | 0.0153 (5) | 0.0014 (5)  |
| O1  | 0.0306 (6)  | 0.0255 (6)  | 0.0223 (5)  | 0.0062 (4)  | 0.0190 (5) | 0.0016 (4)  |
| O2  | 0.0388 (6)  | 0.0192 (5)  | 0.0459 (7)  | -0.0038 (4) | 0.0348 (6) | -0.0010 (5) |
| O3  | 0.0619 (9)  | 0.0373 (7)  | 0.0955 (11) | 0.0094 (6)  | 0.0690 (9) | 0.0134 (7)  |

*Geometric parameters (Å, °)*

|       |             |         |             |
|-------|-------------|---------|-------------|
| C1—N1 | 1.4705 (17) | C10—C11 | 1.5191 (19) |
| C1—C2 | 1.520 (2)   | C10—C13 | 1.525 (2)   |

|            |             |               |             |
|------------|-------------|---------------|-------------|
| C1—H1A     | 0.9900      | C10—H10       | 1.0000      |
| C1—H1B     | 0.9900      | C11—C12       | 1.5050 (19) |
| C2—C3      | 1.5101 (19) | C11—H11       | 1.0000      |
| C2—C11     | 1.5252 (18) | C12—O1        | 1.2426 (16) |
| C2—H2      | 1.0000      | C12—N1        | 1.3402 (17) |
| C3—C4      | 1.514 (2)   | C13—O3        | 1.2005 (17) |
| C3—H3A     | 0.9900      | C13—O2        | 1.3154 (17) |
| C3—H3B     | 0.9900      | C14—N1        | 1.4666 (19) |
| C4—C5      | 1.398 (2)   | C14—C16       | 1.512 (2)   |
| C4—C9      | 1.4067 (19) | C14—C15       | 1.515 (2)   |
| C5—C6      | 1.382 (2)   | C14—H10A      | 1.0000      |
| C5—H5      | 0.9500      | C15—H15A      | 0.9800      |
| C6—C7      | 1.384 (2)   | C15—H15B      | 0.9800      |
| C6—H6      | 0.9500      | C15—H15C      | 0.9800      |
| C7—C8      | 1.386 (2)   | C16—H16A      | 0.9800      |
| C7—H7      | 0.9500      | C16—H16B      | 0.9800      |
| C8—C9      | 1.395 (2)   | C16—H16C      | 0.9800      |
| C8—H8      | 0.9500      | O2—H2O        | 0.8567      |
| C9—C10     | 1.5358 (19) |               |             |
|            |             |               |             |
| N1—C1—C2   | 101.87 (11) | C13—C10—C9    | 109.95 (11) |
| N1—C1—H1A  | 111.4       | C11—C10—H10   | 107.6       |
| C2—C1—H1A  | 111.4       | C13—C10—H10   | 107.6       |
| N1—C1—H1B  | 111.4       | C9—C10—H10    | 107.6       |
| C2—C1—H1B  | 111.4       | C12—C11—C10   | 120.47 (11) |
| H1A—C1—H1B | 109.3       | C12—C11—C2    | 102.35 (11) |
| C3—C2—C1   | 119.24 (11) | C10—C11—C2    | 114.23 (11) |
| C3—C2—C11  | 110.09 (11) | C12—C11—H11   | 106.3       |
| C1—C2—C11  | 101.86 (11) | C10—C11—H11   | 106.3       |
| C3—C2—H2   | 108.4       | C2—C11—H11    | 106.3       |
| C1—C2—H2   | 108.4       | O1—C12—N1     | 124.96 (13) |
| C11—C2—H2  | 108.4       | O1—C12—C11    | 127.15 (12) |
| C2—C3—C4   | 110.29 (11) | N1—C12—C11    | 107.83 (11) |
| C2—C3—H3A  | 109.6       | O3—C13—O2     | 123.31 (14) |
| C4—C3—H3A  | 109.6       | O3—C13—C10    | 123.27 (13) |
| C2—C3—H3B  | 109.6       | O2—C13—C10    | 113.41 (11) |
| C4—C3—H3B  | 109.6       | N1—C14—C16    | 111.31 (13) |
| H3A—C3—H3B | 108.1       | N1—C14—C15    | 109.98 (14) |
| C5—C4—C9   | 118.36 (14) | C16—C14—C15   | 112.27 (15) |
| C5—C4—C3   | 118.63 (12) | N1—C14—H10A   | 107.7       |
| C9—C4—C3   | 122.98 (12) | C16—C14—H10A  | 107.7       |
| C6—C5—C4   | 121.76 (14) | C15—C14—H10A  | 107.7       |
| C6—C5—H5   | 119.1       | C14—C15—H15A  | 109.5       |
| C4—C5—H5   | 119.1       | C14—C15—H15B  | 109.5       |
| C5—C6—C7   | 119.71 (14) | H15A—C15—H15B | 109.5       |
| C5—C6—H6   | 120.1       | C14—C15—H15C  | 109.5       |
| C7—C6—H6   | 120.1       | H15A—C15—H15C | 109.5       |
| C6—C7—C8   | 119.56 (15) | H15B—C15—H15C | 109.5       |

|                 |              |                |              |
|-----------------|--------------|----------------|--------------|
| C6—C7—H7        | 120.2        | C14—C16—H16A   | 109.5        |
| C8—C7—H7        | 120.2        | C14—C16—H16B   | 109.5        |
| C7—C8—C9        | 121.35 (14)  | H16A—C16—H16B  | 109.5        |
| C7—C8—H8        | 119.3        | C14—C16—H16C   | 109.5        |
| C9—C8—H8        | 119.3        | H16A—C16—H16C  | 109.5        |
| C8—C9—C4        | 119.25 (13)  | H16B—C16—H16C  | 109.5        |
| C8—C9—C10       | 117.75 (12)  | C12—N1—C14     | 123.11 (12)  |
| C4—C9—C10       | 123.00 (13)  | C12—N1—C1      | 112.46 (12)  |
| C11—C10—C13     | 114.78 (11)  | C14—N1—C1      | 123.31 (12)  |
| C11—C10—C9      | 108.97 (11)  | C13—O2—H2O     | 108.7        |
| N1—C1—C2—C3     | -155.24 (12) | C9—C10—C11—C2  | 44.17 (15)   |
| N1—C1—C2—C11    | -33.92 (13)  | C3—C2—C11—C12  | 162.67 (11)  |
| C1—C2—C3—C4     | 166.66 (12)  | C1—C2—C11—C12  | 35.21 (13)   |
| C11—C2—C3—C4    | 49.57 (15)   | C3—C2—C11—C10  | -65.43 (15)  |
| C2—C3—C4—C5     | 161.10 (12)  | C1—C2—C11—C10  | 167.11 (11)  |
| C2—C3—C4—C9     | -21.14 (18)  | C10—C11—C12—O1 | 31.0 (2)     |
| C9—C4—C5—C6     | -0.7 (2)     | C2—C11—C12—O1  | 159.03 (14)  |
| C3—C4—C5—C6     | 177.20 (13)  | C10—C11—C12—N1 | -151.79 (12) |
| C4—C5—C6—C7     | -0.4 (2)     | C2—C11—C12—N1  | -23.74 (14)  |
| C5—C6—C7—C8     | 0.9 (2)      | C11—C10—C13—O3 | -140.85 (16) |
| C6—C7—C8—C9     | -0.2 (2)     | C9—C10—C13—O3  | 95.89 (18)   |
| C7—C8—C9—C4     | -0.9 (2)     | C11—C10—C13—O2 | 39.50 (17)   |
| C7—C8—C9—C10    | 179.68 (13)  | C9—C10—C13—O2  | -83.76 (14)  |
| C5—C4—C9—C8     | 1.3 (2)      | O1—C12—N1—C14  | 10.9 (2)     |
| C3—C4—C9—C8     | -176.47 (13) | C11—C12—N1—C14 | -166.44 (12) |
| C5—C4—C9—C10    | -179.29 (13) | O1—C12—N1—C1   | 179.09 (13)  |
| C3—C4—C9—C10    | 3.0 (2)      | C11—C12—N1—C1  | 1.79 (16)    |
| C8—C9—C10—C11   | 165.75 (12)  | C16—C14—N1—C12 | -124.19 (15) |
| C4—C9—C10—C11   | -13.68 (18)  | C15—C14—N1—C12 | 110.74 (16)  |
| C8—C9—C10—C13   | -67.65 (16)  | C16—C14—N1—C1  | 68.85 (18)   |
| C4—C9—C10—C13   | 112.92 (14)  | C15—C14—N1—C1  | -56.23 (18)  |
| C13—C10—C11—C12 | 42.86 (17)   | C2—C1—N1—C12   | 20.97 (15)   |
| C9—C10—C11—C12  | 166.65 (11)  | C2—C1—N1—C14   | -170.83 (12) |
| C13—C10—C11—C2  | -79.61 (15)  |                |              |

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$Cg3$  is the centroid of the C4—C9 rings.

| $D—H\cdots A$                      | $D—H$ | $H\cdots A$ | $D\cdots A$ | $D—H\cdots A$ |
|------------------------------------|-------|-------------|-------------|---------------|
| O2—H2O $\cdots$ O1 <sup>i</sup>    | 0.86  | 1.74        | 2.5885 (17) | 171           |
| C2—H2 $\cdots$ O2                  | 1.00  | 2.33        | 3.002 (2)   | 124           |
| C3—H3B $\cdots$ O2 <sup>ii</sup>   | 0.99  | 2.56        | 3.500 (2)   | 159           |
| C16—H16A $\cdots$ O3 <sup>i</sup>  | 0.98  | 2.55        | 3.531 (3)   | 176           |
| C3—H3A $\cdots$ Cg3 <sup>iii</sup> | 0.99  | 2.68        | 3.6164 (16) | 158           |

Symmetry codes: (i)  $-x+3/2, y+1/2, -z+1/2$ ; (ii)  $-x+3/2, -y+3/2, -z+1$ ; (iii)  $-x+3/2, -y+1/2, -z+1$ .

(4*RS*)-3-Oxo-2-phenyl-2,3,4,9-tetrahydro-1*H*-benzo[*f*]isoindole-4-carboxylic acid (III)

## Crystal data

|                                |   |
|--------------------------------|---|
| $C_{19}H_{15}NO_3$             | $F(000) = 640$  |
| $M_r = 305.32$                 | $D_x = 1.431 \text{ Mg m}^{-3}$                         |
| Monoclinic, $P2_1/c$           | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| $a = 11.9915 (10) \text{ \AA}$ | Cell parameters from 1457 reflections                   |
| $b = 7.1296 (6) \text{ \AA}$   | $\theta = 3.2\text{--}27.5^\circ$                       |
| $c = 16.7967 (16) \text{ \AA}$ | $\mu = 0.10 \text{ mm}^{-1}$                            |
| $\beta = 99.338 (5)^\circ$     | $T = 100 \text{ K}$                                     |
| $V = 1417.0 (2) \text{ \AA}^3$ | Needle, colourless                                      |
| $Z = 4$                        | $0.40 \times 0.04 \times 0.03 \text{ mm}$               |

## Data collection

|   |  |
|---|--|
| Bruker Kappa APEXII area-detector diffractometer                        | 3226 independent reflections   |
| $\varphi$ and $\omega$ scans  | 1938 reflections with $I > 2\sigma(I)$                                 |
| Absorption correction: multi-scan (SADABS; Krause <i>et al.</i> , 2015) | $R_{\text{int}} = 0.088$   |
| $T_{\text{min}} = 0.842$ , $T_{\text{max}} = 1.000$                     | $\theta_{\text{max}} = 27.8^\circ$ , $\theta_{\text{min}} = 3.1^\circ$ |
| 13142 measured reflections  | $h = -15 \rightarrow 15$   |
|   | $k = -9 \rightarrow 8$   |
|   | $l = -21 \rightarrow 21$   |

## Refinement

|                                  |  |
|----------------------------------|--|
| Refinement on $F^2$              | Secondary atom site location: difference Fourier map     |
| Least-squares matrix: full       | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.054$  | H-atom parameters constrained                            |
| $wR(F^2) = 0.128$                | $w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 0.4913P]$        |
| $S = 1.00$                       | where $P = (F_o^2 + 2F_c^2)/3$                           |
| 3226 reflections                 | $(\Delta/\sigma)_{\text{max}} < 0.001$                   |
| 209 parameters                   | $\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$      |
| 0 restraints                     | $\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$     |
| Primary atom site location: dual |  |

## 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 ( $\text{\AA}^2$ )

|     | <i>x</i>     | <i>y</i>   | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|------------|--------------|----------------------------------|
| C1  | 0.67655 (19) | 0.6704 (3) | 0.52923 (14) | 0.0135 (5)                       |
| H1A | 0.677388     | 0.550199   | 0.499836     | 0.016*                           |
| H1B | 0.715759     | 0.767277   | 0.501825     | 0.016*                           |
| C2  | 0.55840 (19) | 0.7292 (3) | 0.53520 (14) | 0.0121 (5)                       |
| C3  | 0.46823 (19) | 0.7638 (3) | 0.46488 (13) | 0.0121 (5)                       |
| H3A | 0.455998     | 0.648343   | 0.431850     | 0.015*                           |
| H3B | 0.493059     | 0.863982   | 0.430895     | 0.015*                           |
| C4  | 0.35804 (19) | 0.8212 (3) | 0.49095 (14) | 0.0120 (5)                       |
| C5  | 0.2653 (2)   | 0.8560 (3) | 0.43085 (15) | 0.0145 (5)                       |

|     |              |            |              |            |
|-----|--------------|------------|--------------|------------|
| H5  | 0.273609     | 0.843410   | 0.375808     | 0.017*     |
| C6  | 0.1616 (2)   | 0.9085 (3) | 0.44982 (15) | 0.0169 (5) |
| H6  | 0.098667     | 0.927258   | 0.408338     | 0.020*     |
| C7  | 0.1507 (2)   | 0.9336 (3) | 0.53027 (15) | 0.0200 (6) |
| H7  | 0.080529     | 0.972954   | 0.543958     | 0.024*     |
| C8  | 0.2417 (2)   | 0.9012 (3) | 0.59016 (15) | 0.0192 (6) |
| H8  | 0.233665     | 0.919964   | 0.644939     | 0.023*     |
| C9  | 0.34539 (19) | 0.8413 (3) | 0.57183 (14) | 0.0133 (5) |
| C10 | 0.44045 (18) | 0.8008 (3) | 0.64234 (14) | 0.0136 (5) |
| H10 | 0.455695     | 0.918657   | 0.674566     | 0.016*     |
| C11 | 0.54682 (19) | 0.7469 (3) | 0.61223 (14) | 0.0123 (5) |
| C12 | 0.65455 (19) | 0.7023 (3) | 0.66446 (14) | 0.0131 (5) |
| C13 | 0.40316 (19) | 0.6495 (3) | 0.69763 (14) | 0.0154 (5) |
| C14 | 0.83942 (19) | 0.5785 (3) | 0.63723 (14) | 0.0137 (5) |
| C15 | 0.92114 (19) | 0.6081 (3) | 0.58824 (15) | 0.0155 (5) |
| H15 | 0.903718     | 0.680544   | 0.540364     | 0.019*     |
| C16 | 1.0281 (2)   | 0.5312 (3) | 0.60984 (16) | 0.0193 (6) |
| H16 | 1.083583     | 0.551149   | 0.576249     | 0.023*     |
| C17 | 1.0553 (2)   | 0.4258 (3) | 0.67955 (16) | 0.0219 (6) |
| H17 | 1.129055     | 0.375083   | 0.694328     | 0.026*     |
| C18 | 0.9731 (2)   | 0.3957 (3) | 0.72738 (15) | 0.0200 (6) |
| H18 | 0.990690     | 0.323139   | 0.775217     | 0.024*     |
| C19 | 0.8661 (2)   | 0.4695 (3) | 0.70646 (15) | 0.0178 (6) |
| H19 | 0.810335     | 0.445974   | 0.739447     | 0.021*     |
| N1  | 0.72847 (16) | 0.6501 (3) | 0.61417 (11) | 0.0140 (4) |
| O1  | 0.67475 (14) | 0.7100 (2) | 0.73924 (10) | 0.0175 (4) |
| O2  | 0.39637 (15) | 0.4801 (2) | 0.66416 (10) | 0.0210 (4) |
| H2O | 0.377299     | 0.401658   | 0.696782     | 0.031*     |
| O3  | 0.38111 (16) | 0.6825 (2) | 0.76358 (11) | 0.0279 (5) |

Atomic displacement parameters ( $\text{\AA}^2$ )

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1  | 0.0167 (12) | 0.0128 (12) | 0.0107 (12) | −0.0027 (9)  | 0.0017 (10)  | 0.0010 (9)   |
| C2  | 0.0155 (12) | 0.0065 (11) | 0.0147 (13) | −0.0011 (9)  | 0.0036 (10)  | 0.0014 (9)   |
| C3  | 0.0152 (12) | 0.0122 (11) | 0.0088 (12) | 0.0007 (9)   | 0.0012 (9)   | 0.0012 (9)   |
| C4  | 0.0136 (12) | 0.0087 (11) | 0.0135 (12) | −0.0026 (9)  | 0.0014 (9)   | −0.0003 (9)  |
| C5  | 0.0185 (13) | 0.0126 (12) | 0.0124 (12) | −0.0031 (10) | 0.0032 (10)  | 0.0002 (10)  |
| C6  | 0.0159 (13) | 0.0169 (13) | 0.0163 (14) | −0.0009 (10) | −0.0023 (10) | 0.0024 (10)  |
| C7  | 0.0131 (13) | 0.0285 (15) | 0.0196 (14) | 0.0019 (10)  | 0.0056 (11)  | 0.0051 (11)  |
| C8  | 0.0183 (13) | 0.0257 (14) | 0.0142 (13) | 0.0010 (10)  | 0.0048 (11)  | 0.0022 (10)  |
| C9  | 0.0147 (12) | 0.0117 (11) | 0.0139 (12) | −0.0006 (9)  | 0.0032 (10)  | 0.0008 (10)  |
| C10 | 0.0138 (12) | 0.0159 (12) | 0.0111 (12) | 0.0000 (9)   | 0.0024 (10)  | 0.0016 (9)   |
| C11 | 0.0153 (12) | 0.0114 (12) | 0.0100 (12) | −0.0011 (9)  | 0.0012 (10)  | 0.0008 (9)   |
| C12 | 0.0140 (12) | 0.0116 (12) | 0.0138 (13) | −0.0015 (9)  | 0.0030 (10)  | 0.0002 (9)   |
| C13 | 0.0119 (12) | 0.0215 (13) | 0.0124 (13) | 0.0026 (9)   | 0.0006 (10)  | 0.0015 (10)  |
| C14 | 0.0127 (12) | 0.0123 (12) | 0.0151 (13) | 0.0000 (9)   | −0.0008 (10) | −0.0011 (10) |
| C15 | 0.0181 (13) | 0.0159 (12) | 0.0129 (13) | −0.0002 (10) | 0.0035 (10)  | 0.0013 (10)  |

|     |             |             |             |              |              |              |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C16 | 0.0167 (13) | 0.0187 (13) | 0.0237 (14) | -0.0008 (10) | 0.0068 (11)  | -0.0024 (11) |
| C17 | 0.0163 (13) | 0.0217 (14) | 0.0260 (15) | 0.0055 (10)  | -0.0016 (11) | -0.0015 (11) |
| C18 | 0.0212 (14) | 0.0221 (13) | 0.0152 (13) | 0.0044 (10)  | -0.0011 (11) | 0.0045 (11)  |
| C19 | 0.0176 (13) | 0.0209 (14) | 0.0161 (13) | -0.0011 (10) | 0.0057 (11)  | 0.0011 (10)  |
| N1  | 0.0136 (10) | 0.0173 (10) | 0.0109 (10) | -0.0003 (8)  | 0.0016 (8)   | 0.0004 (8)   |
| O1  | 0.0185 (9)  | 0.0216 (9)  | 0.0123 (9)  | 0.0023 (7)   | 0.0021 (7)   | -0.0017 (7)  |
| O2  | 0.0311 (11) | 0.0161 (9)  | 0.0173 (10) | -0.0027 (8)  | 0.0084 (8)   | 0.0028 (7)   |
| O3  | 0.0397 (12) | 0.0307 (11) | 0.0168 (10) | 0.0002 (9)   | 0.0148 (9)   | -0.0007 (8)  |

*Geometric parameters (Å, °)*

|            |             |             |             |
|------------|-------------|-------------|-------------|
| C1—N1      | 1.468 (3)   | C10—C13     | 1.537 (3)   |
| C1—C2      | 1.496 (3)   | C10—H10     | 1.0000      |
| C1—H1A     | 0.9900      | C11—C12     | 1.474 (3)   |
| C1—H1B     | 0.9900      | C12—O1      | 1.241 (3)   |
| C2—C11     | 1.329 (3)   | C12—N1      | 1.371 (3)   |
| C2—C3      | 1.487 (3)   | C13—O3      | 1.203 (3)   |
| C3—C4      | 1.514 (3)   | C13—O2      | 1.330 (3)   |
| C3—H3A     | 0.9900      | C14—C19     | 1.392 (3)   |
| C3—H3B     | 0.9900      | C14—C15     | 1.394 (3)   |
| C4—C5      | 1.398 (3)   | C14—N1      | 1.419 (3)   |
| C4—C9      | 1.398 (3)   | C15—C16     | 1.388 (3)   |
| C5—C6      | 1.384 (3)   | C15—H15     | 0.9500      |
| C5—H5      | 0.9500      | C16—C17     | 1.385 (4)   |
| C6—C7      | 1.390 (3)   | C16—H16     | 0.9500      |
| C6—H6      | 0.9500      | C17—C18     | 1.386 (4)   |
| C7—C8      | 1.378 (3)   | C17—H17     | 0.9500      |
| C7—H7      | 0.9500      | C18—C19     | 1.379 (3)   |
| C8—C9      | 1.396 (3)   | C18—H18     | 0.9500      |
| C8—H8      | 0.9500      | C19—H19     | 0.9500      |
| C9—C10     | 1.532 (3)   | O2—H2O      | 0.8400      |
| C10—C11    | 1.497 (3)   |             |             |
| N1—C1—C2   | 102.67 (18) | C9—C10—C13  | 110.27 (19) |
| N1—C1—H1A  | 111.2       | C11—C10—H10 | 107.9       |
| C2—C1—H1A  | 111.2       | C9—C10—H10  | 107.9       |
| N1—C1—H1B  | 111.2       | C13—C10—H10 | 107.9       |
| C2—C1—H1B  | 111.2       | C2—C11—C12  | 109.8 (2)   |
| H1A—C1—H1B | 109.1       | C2—C11—C10  | 125.6 (2)   |
| C11—C2—C3  | 125.5 (2)   | C12—C11—C10 | 124.5 (2)   |
| C11—C2—C1  | 110.0 (2)   | O1—C12—N1   | 126.6 (2)   |
| C3—C2—C1   | 124.6 (2)   | O1—C12—C11  | 127.0 (2)   |
| C2—C3—C4   | 111.79 (19) | N1—C12—C11  | 106.48 (19) |
| C2—C3—H3A  | 109.3       | O3—C13—O2   | 124.0 (2)   |
| C4—C3—H3A  | 109.3       | O3—C13—C10  | 123.3 (2)   |
| C2—C3—H3B  | 109.3       | O2—C13—C10  | 112.72 (19) |
| C4—C3—H3B  | 109.3       | C19—C14—C15 | 119.3 (2)   |
| H3A—C3—H3B | 107.9       | C19—C14—N1  | 120.5 (2)   |

|                |              |                 |             |
|----------------|--------------|-----------------|-------------|
| C5—C4—C9       | 119.0 (2)    | C15—C14—N1      | 120.1 (2)   |
| C5—C4—C3       | 117.9 (2)    | C16—C15—C14     | 119.6 (2)   |
| C9—C4—C3       | 123.1 (2)    | C16—C15—H15     | 120.2       |
| C6—C5—C4       | 121.4 (2)    | C14—C15—H15     | 120.2       |
| C6—C5—H5       | 119.3        | C17—C16—C15     | 121.1 (2)   |
| C4—C5—H5       | 119.3        | C17—C16—H16     | 119.5       |
| C5—C6—C7       | 119.2 (2)    | C15—C16—H16     | 119.5       |
| C5—C6—H6       | 120.4        | C16—C17—C18     | 118.8 (2)   |
| C7—C6—H6       | 120.4        | C16—C17—H17     | 120.6       |
| C8—C7—C6       | 120.0 (2)    | C18—C17—H17     | 120.6       |
| C8—C7—H7       | 120.0        | C19—C18—C17     | 120.9 (2)   |
| C6—C7—H7       | 120.0        | C19—C18—H18     | 119.6       |
| C7—C8—C9       | 121.2 (2)    | C17—C18—H18     | 119.6       |
| C7—C8—H8       | 119.4        | C18—C19—C14     | 120.3 (2)   |
| C9—C8—H8       | 119.4        | C18—C19—H19     | 119.9       |
| C8—C9—C4       | 119.1 (2)    | C14—C19—H19     | 119.9       |
| C8—C9—C10      | 117.7 (2)    | C12—N1—C14      | 126.9 (2)   |
| C4—C9—C10      | 123.2 (2)    | C12—N1—C1       | 110.92 (18) |
| C11—C10—C9     | 110.81 (19)  | C14—N1—C1       | 122.12 (18) |
| C11—C10—C13    | 111.80 (18)  | C13—O2—H2O      | 109.5       |
| N1—C1—C2—C11   | 2.2 (2)      | C13—C10—C11—C12 | -57.7 (3)   |
| N1—C1—C2—C3    | -178.22 (19) | C2—C11—C12—O1   | 177.6 (2)   |
| C11—C2—C3—C4   | -0.6 (3)     | C10—C11—C12—O1  | -3.2 (4)    |
| C1—C2—C3—C4    | 179.9 (2)    | C2—C11—C12—N1   | -2.4 (3)    |
| C2—C3—C4—C5    | -179.24 (19) | C10—C11—C12—N1  | 176.7 (2)   |
| C2—C3—C4—C9    | 1.0 (3)      | C11—C10—C13—O3  | 128.6 (2)   |
| C9—C4—C5—C6    | -0.4 (3)     | C9—C10—C13—O3   | -107.7 (3)  |
| C3—C4—C5—C6    | 179.8 (2)    | C11—C10—C13—O2  | -52.4 (3)   |
| C4—C5—C6—C7    | 2.3 (3)      | C9—C10—C13—O2   | 71.4 (2)    |
| C5—C6—C7—C8    | -1.7 (4)     | C19—C14—C15—C16 | -1.0 (4)    |
| C6—C7—C8—C9    | -0.7 (4)     | N1—C14—C15—C16  | -177.5 (2)  |
| C7—C8—C9—C4    | 2.6 (4)      | C14—C15—C16—C17 | -0.3 (4)    |
| C7—C8—C9—C10   | -177.5 (2)   | C15—C16—C17—C18 | 0.9 (4)     |
| C5—C4—C9—C8    | -2.0 (3)     | C16—C17—C18—C19 | -0.3 (4)    |
| C3—C4—C9—C8    | 177.8 (2)    | C17—C18—C19—C14 | -1.0 (4)    |
| C5—C4—C9—C10   | 178.1 (2)    | C15—C14—C19—C18 | 1.6 (4)     |
| C3—C4—C9—C10   | -2.2 (3)     | N1—C14—C19—C18  | 178.1 (2)   |
| C8—C9—C10—C11  | -177.4 (2)   | O1—C12—N1—C14   | 6.1 (4)     |
| C4—C9—C10—C11  | 2.5 (3)      | C11—C12—N1—C14  | -173.8 (2)  |
| C8—C9—C10—C13  | 58.3 (3)     | O1—C12—N1—C1    | -176.2 (2)  |
| C4—C9—C10—C13  | -121.8 (2)   | C11—C12—N1—C1   | 3.9 (2)     |
| C3—C2—C11—C12  | -179.5 (2)   | C19—C14—N1—C12  | 32.1 (3)    |
| C1—C2—C11—C12  | 0.0 (3)      | C15—C14—N1—C12  | -151.4 (2)  |
| C3—C2—C11—C10  | 1.4 (4)      | C19—C14—N1—C1   | -145.4 (2)  |
| C1—C2—C11—C10  | -179.1 (2)   | C15—C14—N1—C1   | 31.0 (3)    |
| C9—C10—C11—C2  | -2.1 (3)     | C2—C1—N1—C12    | -3.8 (2)    |
| C13—C10—C11—C2 | 121.3 (2)    | C2—C1—N1—C14    | 174.04 (19) |

C9—C10—C11—C12 178.9 (2)

*Hydrogen-bond geometry* ( $\text{\AA}$ ,  $^\circ$ )

Cg1, Cg2, Cg3 and Cg4 are the centroids of the N1/C1/C2/C11/C12, C2—C11 C4—C9 and C16—C21 rings, respectively.

| <i>D</i> —H $\cdots$ <i>A</i>      | <i>D</i> —H | H $\cdots$ <i>A</i> | <i>D</i> $\cdots$ <i>A</i> | <i>D</i> —H $\cdots$ <i>A</i> |
|------------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O2—H2O $\cdots$ O1 <sup>i</sup>    | 0.84        | 1.90                | 2.741 (2)                  | 174                           |
| C1—H1A $\cdots$ Cg2 <sup>ii</sup>  | 0.99        | 2.91                | 3.756 (2)                  | 144                           |
| C1—H1B $\cdots$ Cg3 <sup>iii</sup> | 0.99        | 2.58                | 3.427 (2)                  | 144                           |
| C3—H3A $\cdots$ Cg1 <sup>ii</sup>  | 0.99        | 2.70                | 3.593 (2)                  | 150                           |
| C3—H3B $\cdots$ Cg2 <sup>iii</sup> | 0.99        | 2.60                | 3.388 (2)                  | 137                           |
| C18—H18 $\cdots$ Cg4 <sup>iv</sup> | 0.95        | 2.61                | 3.452 (2)                  | 148                           |

Symmetry codes: (i)  $-x+1, y-1/2, -z+3/2$ ; (ii)  $-x+1, -y+1, -z+1$ ; (iii)  $-x+1, -y+2, -z+1$ ; (iv)  $-x+2, y-1/2, -z+3/2$ .