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Hydrogen bonding in cyclic imides and amide carboxylic acid derivatives from the facile reaction of *cis*-cyclohexane-1,2-carboxylic anhydride with *o*- and *p*-anisidine and *m*- and *p*-aminobenzoic acids

Graham Smith* and Urs D. Wermuth

Science and Engineering Faculty, Queensland University of Technology, GPO Box 2434, Brisbane, Queensland 4001, Australia
Correspondence e-mail: g.smith@qut.edu.au

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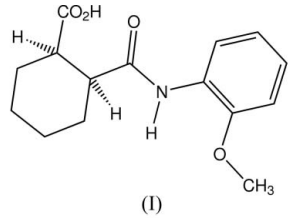
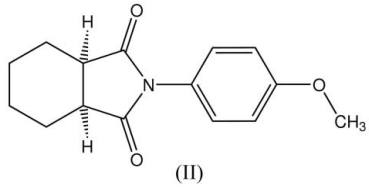
The structures of the open-chain amide carboxylic acid *rac*-*cis*-2-[(2-methoxyphenyl)carbamoyl]cyclohexane-1-carboxylic acid, $C_{15}H_{19}NO_4$, (I), and the cyclic imides *rac*-*cis*-2-(4-methoxyphenyl)-3a,4,5,6,7,7a-hexahydroisoindole-1,3-dione, $C_{15}H_{17}NO_3$, (II), chiral *cis*-3-(1,3-dioxo-3a,4,5,6,7,7a-hexahydroisoindol-2-yl)benzoic acid, $C_{15}H_{15}NO_4$, (III), and *rac*-*cis*-4-(1,3-dioxo-3a,4,5,6,7,7a-hexahydroisoindol-2-yl)benzoic acid monohydrate, $C_{15}H_{15}NO_4\cdot H_2O$, (IV), are reported. In the amide acid (I), the phenylcarbamoyl group is essentially planar [maximum deviation from the least-squares plane = 0.060 (1) Å for the amide O atom] and the molecules form discrete centrosymmetric dimers through intermolecular cyclic carboxy–carboxy O—H···O hydrogen-bonding interactions [graph-set notation $R_2^2(8)$]. The cyclic imides (II)–(IV) are conformationally similar, with comparable benzene ring rotations about the imide N—C_{ar} bond [dihedral angles between the benzene and isoindole rings = 51.55 (7)° in (II), 59.22 (12)° in (III) and 51.99 (14)° in (IV)]. Unlike (II), in which only weak intermolecular C—H···O_{imide} hydrogen bonding is present, the crystal packing of imides (III) and (IV) shows strong intermolecular carboxylic acid O—H···O hydrogen-bonding associations. With (III), these involve imide O-atom acceptors, giving one-dimensional zigzag chains [graph-set $C(9)$], while with the monohydrate (IV), the hydrogen bond involves the partially disordered water molecule which also bridges molecules through both imide and carboxy O-atom acceptors in a cyclic $R_4^4(12)$ association, giving a two-dimensional sheet structure. The structures reported here expand the structural database for compounds of this series formed from the facile reaction of *cis*-cyclohexane-1,2-dicarboxylic anhydride with substituted anilines, in which there is a much larger incidence of cyclic imides compared to amide carboxylic acids.

Comment

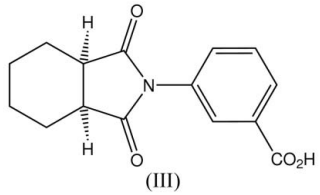
The 1:1 stoichiometric reaction of *cis*-cyclohexane-1,2-dicarboxylic anhydride (*cis*-CHDC anhydride) with substituted anilines has been found to give both open-chain amide carboxylic acids or more commonly cyclic imides under mild reaction conditions (Smith & Wermuth, 2012*a*). These products are analogous to the phthalanilic acids and phthalimides formed in the reactions of phthalic anhydride with anilines (Perry & Parveen, 2001). We previously reported the structure of the amide acid from the reaction of *cis*-CHDC anhydride with 3-fluoroaniline and the isomeric cyclic imides from the parallel reactions with 2- and 4-fluoroaniline (Smith & Wermuth, 2012*a*), which are among only the very few crystallographically characterized examples of these compounds.



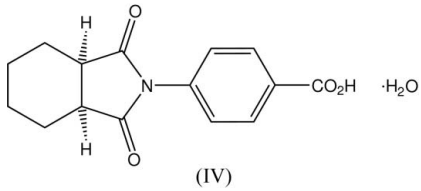
(I)



(II)



(III)



(IV)

The parallel reaction of 2-methoxyaniline (*o*-anisidine), 4-methoxyaniline (*p*-anisidine), 3-carboxyaniline (*m*-aminobenzoic acid) and 4-carboxyaniline (*p*-aminobenzoic acid) with *cis*-CHDC anhydride under common mild reaction conditions in 50% ethanol–water solution yielded, respectively, the open-chain amide carboxylic acid racemic *cis*-2-[(2-methoxyphenyl)carbamoyl]cyclohexane-1-carboxylic acid, (I), and the cyclic imides racemic 2-(4-methoxyphenyl)-3a,4,5,6,7,7a-hexahydroisoindole-1,3-dione, (II), chiral 3-(1,3-dioxo-3a,4,5,6,7,7a-hexahydroisoindol-2-yl)benzoic acid, (III), and racemic 4-(1,3-dioxo-3a,4,5,6,7,7a-hexahydroisoindol-2-yl)benzoic acid monohydrate, (IV) (Figs. 1–4), and the structures are reported here.

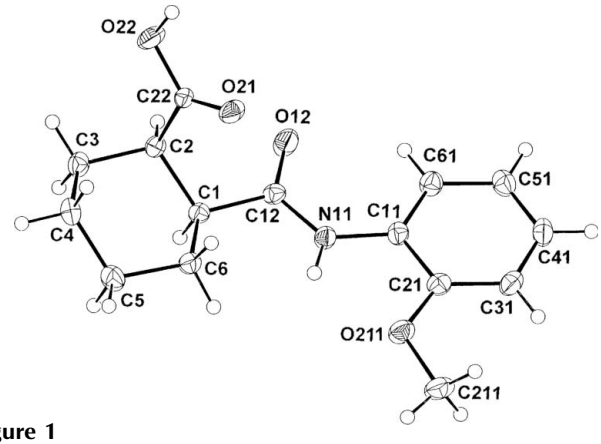


Figure 1
The molecular conformation and atom-labelling scheme for (I). Displacement ellipsoids are drawn at the 40% probability level.

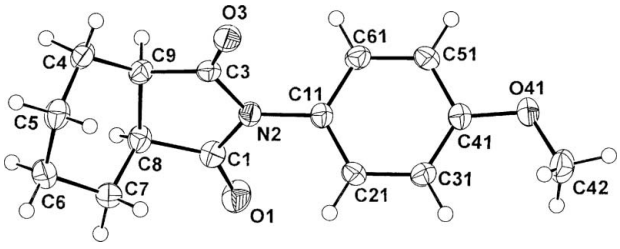


Figure 2
The molecular conformation and atom-labelling scheme for (II). Displacement ellipsoids are drawn at the 40% probability level.

In the racemic amide acid (I) (Fig. 1), the phenylcarbamoyl group is essentially planar [C21—C11—N11—C12 torsion angle = 175.18 (12)°], with a maximum deviation from the least-squares plane of 0.060 (1) Å for the amide O atom. The conformation is stabilized by intramolecular N11—H···O211(methoxy) and aromatic C61—H···O12(carbonyl) interactions [H···O = 2.6003 (14) and 2.8812 (17) Å, respectively]. The carboxy group on the cyclohexane ring is almost parallel to the C1—C3 bond [C1—C2—C22—O22 torsion angle = 173.17 (10)°] and the methoxy group is close to being coplanar with the benzene ring [C11—C21—O211—C211 torsion angle = 173.23 (11)°]. The molecules form discrete centrosymmetric dimers through classical intermolecular cyclic carboxy–carboxy O—H···O hydrogen-bonding interactions (Table 1) [graph-set notation $R_2^2(8)$; Etter *et al.*, 1990] (Fig. 5). The amide group is not involved in intermolecular interactions, which is unlike the 4-chloro-substituted analogue in which the amide group links hydrogen-bonded carboxylic acid chains into a two-dimensional sheet structure (Smith & Wermuth, 2012*b*).

The cyclic imides (II) and (IV) (Figs. 2 and 4) are racemic, while compound (III) is chiral. However, the absolute configurations for the two chiral centres in (III) were not determined, being arbitrarily assigned (C8*S* and C9*R*). Conformationally, compounds (II)–(IV) are similar, with comparable ring rotations about the imide N—C_{ar} bond [minimum torsion angles C1/C3—N2—C11—C21 = −56.72 (18)° for (II), 61.8 (3)° for (III) and −53.8 (3)° for (IV)]. These correspond to dihedral angles of 51.55 (7),

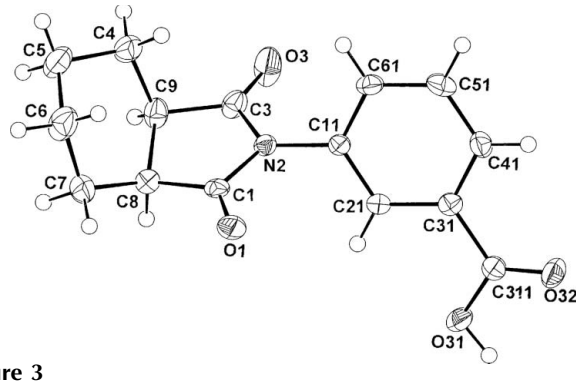


Figure 3
The molecular conformation and atom-labelling scheme for (III). Displacement ellipsoids are drawn at the 40% probability level.

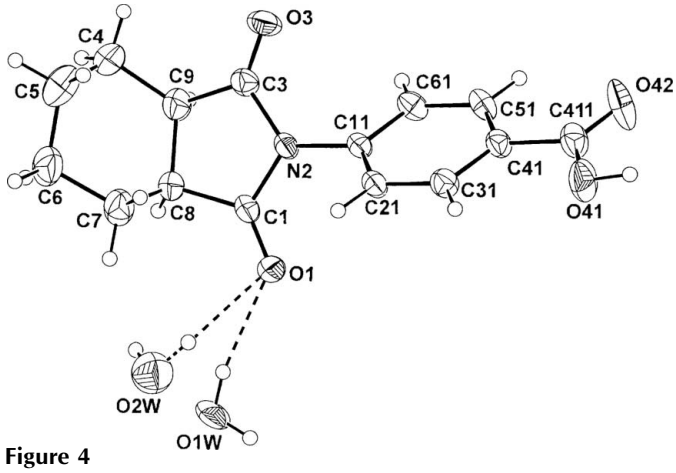


Figure 4
The molecular conformation and atom-labelling scheme for (IV). Displacement ellipsoids are drawn at the 40% probability level. The water molecule of solvation is disordered over two sites [O1*W*, occupancy factor = 0.81 (1); O2*W*, occupancy factor = 0.19 (1)]. Inter-species hydrogen bonds are shown as dashed lines.

59.22 (12) and 51.99 (14)°, respectively, between the benzene ring and the plane of the isoindole ring, in which distortion results in either atom C8 or C9 showing a maximum deviation of 0.157 (1) Å in (II), 0.139 (2) Å in (III) and 0.131 (3) Å in (IV). These values compare closely to those of 0.152 (1) and 0.149 (1) Å for the 2- and 4-fluoro analogues, respectively (Smith & Wermuth, 2012*a*), and 0.153 (3) and 0.138 (4) Å for the 4-bromo- and 3-carboxy-4-hydroxy-substituted analogues, respectively (Smith & Wermuth, 2012*b*). In (III) and (IV), the carboxy substituent groups are close to being coplanar with the attached benzene rings [C21—C31—C311—O32 = −172.2 (2)° in (III) and C31—C41—C411—O42 = −177.3 (3)° in (IV)].

Unlike the structure of (II), in which there are only weak aromatic C—H···O_{imide} hydrogen-bonding interactions (Table 2), the crystal packing of both imides (III) and (IV) shows strong intermolecular O—H···O hydrogen-bonding interactions involving carboxy groups. In (III), these are with imide O-atom acceptors (Table 3), giving one-dimensional zigzag chains [*C*(9)] which extend along (010) (Fig. 6). In the monohydrate (IV), the *para*-carboxy group forms a hydrogen bond with the partially disordered water molecule [O1*W*,

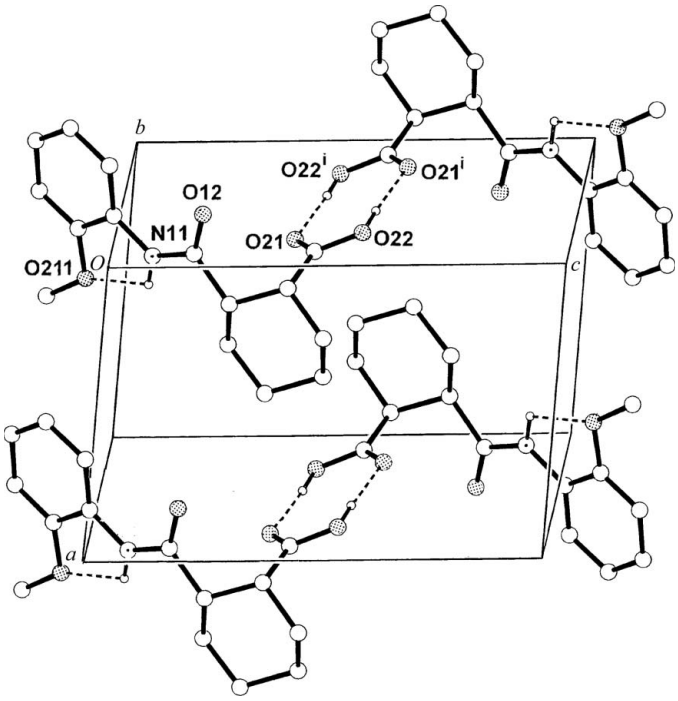


Figure 5
The centrosymmetric hydrogen-bonded dimers in the structure of (I), showing hydrogen-bonding interactions as dashed lines. Non-associative H atoms have been omitted. For symmetry code (i), see Table 1.

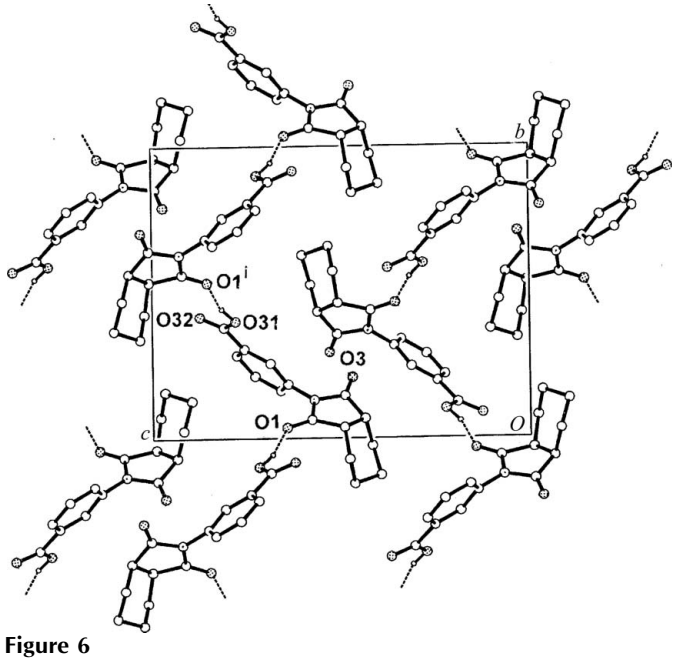


Figure 6
The one-dimensional hydrogen-bonded chain structures in (III), viewed down the *a*-cell direction of the unit cell, showing hydrogen-bonding interactions as dashed lines. Non-associative H atoms have been omitted. For symmetry code (i), see Table 3.

occupancy factor = 0.81 (1); O2*W*, occupancy factor = 0.19 (1)], both components of which also act as donors to both imide and carboxy O-atom acceptors (Table 4). These interactions give an $R_4^4(12)$ ring motif and extend the structure into a two-dimensional sheet (Fig. 7).

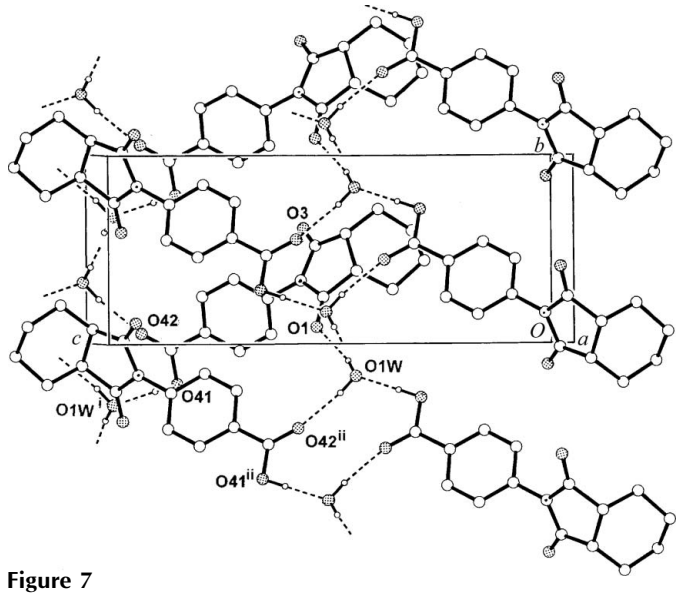


Figure 7
The two-dimensional hydrogen-bonded structure in (IV), viewed approximately down the *a*-cell direction of the unit cell, showing hydrogen-bonding interactions as dashed lines. Non-associative H atoms have been omitted as has the minor-component O2*W* water molecule. For symmetry codes, see Table 4.

The structures reported here expand the structural database for compounds of this series, formed in the facile reaction of *cis*-CHDC anhydride with substituted anilines, among which there is a much larger incidence of cyclic imides compared to amide carboxylic acids [currently 8:3, among known examples, which, apart from our previously reported structures, include the imide from the reaction with 5-benzyloxy-2,4-dichloroaniline (Wang *et al.*, 2005) and those from the reactions with 4-bromoaniline and 5-aminosalicylic acid (Smith & Wermuth, 2012*b*)]. However, there are no apparent structural features which might allow a definitive prediction of the preferred reaction product.

Experimental

The title compounds were synthesized by heating together under reflux for 15 min 1 mmol quantities of *cis*-cyclohexane-1,2-dicarboxylic anhydride and *o*-anisidine [for (I)], *p*-anisidine [for (II)], *m*-aminobenzoic acid [for (III)] and *p*-aminobenzoic acid [for (IV)] in ethanol–water (50 ml, 1:1 *v*/*v*). After volume reduction to 30 ml, the hot-filtered solutions were allowed to evaporate to incipient dryness at room temperature over a period of several weeks, giving either colourless plates [of (I)–(III)] or fine needles [of (IV)] from which specimens were cleaved for structural analyses.

Compound (I)

Crystal data

$C_{15}H_{19}NO_4$
M_r = 277.31
Triclinic, $P\overline{1}$
a = 7.3557 (4) Å
b = 8.3630 (4) Å
c = 11.7128 (6) Å
α = 100.453 (4)°
β = 97.232 (4)°
γ = 104.042 (4)°
V = 676.40 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 200 K
0.45 × 0.30 × 0.18 mm

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

D—H···A	D—H	H···A	D···A	D—H···A
O22—H22···O21^{i}	0.94 (2)	1.76 (2)	2.6866 (14)	173 (2)

Symmetry code: (i) $-x, -y+1, -z+1$.

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.975, T_{max} = 0.980
8079 measured reflections
2640 independent reflections
2082 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.087
S = 1.03
2640 reflections
189 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Compound (II)

Crystal data

$C_{15}H_{17}NO_3$
M_r = 259.30
Monoclinic, $P2_1/n$
a = 11.7119 (5) Å
b = 6.6705 (3) Å
c = 17.2898 (8) Å
β = 109.482 (5)°
V = 1273.42 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 200 K
0.40 × 0.25 × 0.12 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.929, T_{max} = 0.981
8552 measured reflections
2498 independent reflections
1959 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.088
S = 1.09
2498 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Compound (III)

Crystal data

$C_{15}H_{15}NO_4$
M_r = 273.28
Orthorhombic, $P2_12_12_1$
a = 6.4958 (2) Å
b = 12.5236 (4) Å
c = 16.1281 (6) Å
V = 1312.03 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 200 K
0.45 × 0.12 × 0.08 mm

Data collection

Oxford Diffraction Gemini-S Ultra CCD-detector diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.980, T_{max} = 0.990
4693 measured reflections
1728 independent reflections
1395 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

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

D—H···A	D—H	H···A	D···A	D—H···A
C21—H21···O3^{i}	0.93	2.56	3.2205 (15)	128
C51—H51···O1^{ii}	0.93	2.46	3.2914 (17)	149

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O31—H31···O1^{i}	0.89 (3)	1.84 (3)	2.682 (2)	156 (3)

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

Table 4
Hydrogen-bond geometry (Å, °) for (IV).

D—H···A	D—H	H···A	D···A	D—H···A
O41—H41···O1W^{i}	0.90	1.71	2.609 (3)	179
O1W—H11W···O1	0.83	2.08	2.894 (3)	167
O1W—H12W···O42^{ii}	0.82	1.94	2.754 (3)	172
O2W—H21W···O1	0.90	2.10	2.991 (12)	179
O2W—H22W···O42^{iii}	0.90	2.33	3.233 (12)	178

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

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.086
S = 1.10
1728 reflections
185 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Compound (IV)

Crystal data

$C_{15}H_{15}NO_4\cdot H_2O$
M_r = 291.30
Monoclinic, $P2_1/n$
a = 13.077 (2) Å
b = 6.6713 (7) Å
c = 16.432 (2) Å
β = 97.982 (13)°
V = 1419.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 200 K
0.30 × 0.10 × 0.08 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.813, T_{max} = 0.980
9649 measured reflections
2790 independent reflections
1374 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.132
S = 0.85
2790 reflections
194 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Amide and/or carboxylic acid H atoms in (I), (III) and (IV) were located by difference methods and in (I) and (III) both their posi-

tional and isotropic displacement parameters were refined. For (IV), these H atoms were allowed to ride in the refinement, with $U_{iso}(H) = 1.5U_{eq}(O)$. Other H atoms in all structures were allowed to ride in the refinement, with $U_{iso}(H) = 1.2U_{eq}(O)$. Other H atoms in all structures were included in the respective refinements at calculated positions (C—H = 0.93–0.98 Å), with $U_{iso}(H) = 1.2U_{eq}(C)$, using a riding-model approximation. The water molecule of solvation in (IV) was found to be disordered over two adjacent sites [O···O = 1.458 (12) Å], with occupancy factors determined as 0.81 (1) (O1*W*) and 0.19 (1) (O2*W*). The occupancies were subsequently fixed and the minor component was refined isotropically. In chiral (III), in the absence of a suitable heavy atom, Friedel pairs (1062) were merged, the relative configuration of the chiral centres (C8*S* and C9*R*) being arbitrarily assigned.

For all compounds, data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*. Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993) for (I), (III) and (IV); *SHELXS97* (Sheldrick, 2008) for (II). For all compounds, program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 1999); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM3120). Services for accessing these data are described at the back of the journal.

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supplementary materials

Acta Cryst. (2012). C**68**, o327–o331 [doi:10.1107/S0108270112030168]

Hydrogen bonding in cyclic imides and amide carboxylic acid derivatives from the facile reaction of *cis*-cyclohexane-1,2-carboxylic anhydride with *o*- and *p*-anisidine and *m*- and *p*-aminobenzoic acids

Graham Smith and Urs D. Wermuth

(I) 2-[(2-methoxyphenyl)carbamoyl]cyclohexane-1-carboxylic acid

Crystal data

$C_{15}H_{19}NO_4$
M_r = 277.31
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.3557 (4) Å
b = 8.3630 (4) Å
c = 11.7128 (6) Å
α = 100.453 (4)°
β = 97.232 (4)°
γ = 104.042 (4)°
V = 676.40 (6) Å^3
Z = 2
$F(000)$ = 296
D_x = 1.362 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4313 reflections
θ = 3.3–28.8°
μ = 0.10 mm^{-1}
T = 200 K
Flat prism, colourless
0.45 × 0.30 × 0.18 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.975, T_{max} = 0.980
8079 measured reflections
2640 independent reflections
2082 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 26.0°, θ_{min} = 3.3°
h = −9→9
k = −10→10
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.087
S = 1.03
2640 reflections
189 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O12	−0.14926 (13)	0.07111 (14)	0.19578 (9)	0.0449 (4)
O21	0.08493 (13)	0.41630 (11)	0.38713 (8)	0.0321 (3)
O22	0.01483 (14)	0.29940 (13)	0.53760 (8)	0.0375 (4)
O211	0.19154 (12)	0.36885 (11)	−0.06244 (8)	0.0318 (3)
N11	0.04741 (16)	0.20455 (13)	0.08805 (9)	0.0258 (3)
C1	0.18449 (16)	0.13300 (15)	0.26727 (10)	0.0213 (4)
C2	0.13519 (16)	0.14141 (14)	0.39152 (10)	0.0211 (4)
C3	0.30154 (18)	0.12408 (16)	0.47820 (11)	0.0267 (4)
C4	0.48581 (17)	0.25512 (16)	0.47911 (11)	0.0282 (4)
C5	0.53572 (17)	0.24351 (17)	0.35632 (12)	0.0316 (4)
C6	0.37447 (17)	0.25898 (16)	0.26619 (11)	0.0259 (4)
C11	−0.08286 (17)	0.22259 (15)	−0.00513 (10)	0.0235 (4)
C12	0.01120 (17)	0.13474 (15)	0.18109 (11)	0.0236 (4)
C21	−0.00335 (18)	0.31266 (15)	−0.08506 (11)	0.0248 (4)
C22	0.07697 (16)	0.29933 (15)	0.43665 (10)	0.0219 (4)
C31	−0.12003 (19)	0.34059 (16)	−0.17739 (11)	0.0301 (4)
C41	−0.3160 (2)	0.27757 (17)	−0.19104 (12)	0.0322 (4)
C51	−0.39563 (19)	0.18700 (16)	−0.11428 (12)	0.0314 (4)
C61	−0.27918 (18)	0.15905 (16)	−0.02087 (11)	0.0284 (4)
C211	0.2820 (2)	0.44485 (18)	−0.14755 (13)	0.0378 (5)
H1	0.20390	0.02080	0.24310	0.0260*
H2	0.02610	0.04440	0.38650	0.0250*
H3A	0.27110	0.13760	0.55700	0.0320*
H3B	0.31850	0.01190	0.45580	0.0320*
H4A	0.58840	0.23800	0.53200	0.0340*
H4B	0.47270	0.36720	0.50840	0.0340*
H5A	0.56310	0.13610	0.33090	0.0380*
H5B	0.64970	0.33270	0.35930	0.0380*
H6A	0.40640	0.23830	0.18800	0.0310*
H6B	0.36120	0.37300	0.28410	0.0310*
H11	0.164 (2)	0.2508 (18)	0.0824 (13)	0.045 (4)*
H21A	0.41750	0.47850	−0.12170	0.0450*
H21B	0.23900	0.54230	−0.15640	0.0450*
H21C	0.25030	0.36500	−0.22190	0.0450*
H22	−0.015 (3)	0.402 (3)	0.5587 (17)	0.082 (6)*
H31	−0.06730	0.40120	−0.23000	0.0360*
H41	−0.39450	0.29680	−0.25280	0.0390*
H51	−0.52710	0.14440	−0.12470	0.0380*

H61	−0.33300	0.09770	0.03110	0.0340*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O12	0.0229 (5)	0.0766 (8)	0.0336 (6)	0.0013 (5)	0.0032 (4)	0.0261 (5)
O21	0.0487 (6)	0.0297 (5)	0.0297 (5)	0.0212 (4)	0.0179 (4)	0.0142 (4)
O22	0.0604 (7)	0.0392 (6)	0.0276 (6)	0.0274 (5)	0.0234 (5)	0.0153 (4)
O211	0.0295 (5)	0.0369 (5)	0.0288 (5)	0.0041 (4)	0.0088 (4)	0.0113 (4)
N11	0.0217 (6)	0.0309 (6)	0.0247 (6)	0.0042 (5)	0.0033 (5)	0.0102 (5)
C1	0.0237 (6)	0.0200 (6)	0.0205 (7)	0.0071 (5)	0.0046 (5)	0.0031 (5)
C2	0.0231 (6)	0.0189 (6)	0.0214 (7)	0.0040 (5)	0.0043 (5)	0.0069 (5)
C3	0.0336 (7)	0.0246 (7)	0.0248 (7)	0.0118 (6)	0.0040 (5)	0.0084 (5)
C4	0.0280 (7)	0.0273 (7)	0.0285 (8)	0.0097 (6)	−0.0027 (5)	0.0068 (5)
C5	0.0220 (7)	0.0364 (8)	0.0349 (8)	0.0062 (6)	0.0019 (6)	0.0084 (6)
C6	0.0237 (6)	0.0308 (7)	0.0236 (7)	0.0064 (5)	0.0058 (5)	0.0073 (5)
C11	0.0272 (7)	0.0222 (6)	0.0202 (7)	0.0085 (5)	0.0013 (5)	0.0018 (5)
C12	0.0236 (6)	0.0245 (7)	0.0204 (7)	0.0046 (5)	0.0043 (5)	0.0015 (5)
C21	0.0294 (7)	0.0217 (6)	0.0223 (7)	0.0074 (5)	0.0057 (5)	0.0010 (5)
C22	0.0197 (6)	0.0288 (7)	0.0172 (6)	0.0065 (5)	0.0025 (5)	0.0058 (5)
C31	0.0430 (8)	0.0297 (7)	0.0216 (7)	0.0143 (6)	0.0089 (6)	0.0073 (5)
C41	0.0392 (8)	0.0347 (8)	0.0241 (7)	0.0174 (6)	−0.0023 (6)	0.0055 (6)
C51	0.0275 (7)	0.0331 (8)	0.0313 (8)	0.0095 (6)	−0.0005 (6)	0.0037 (6)
C61	0.0287 (7)	0.0282 (7)	0.0278 (8)	0.0060 (6)	0.0040 (6)	0.0083 (6)
C211	0.0412 (8)	0.0416 (9)	0.0365 (9)	0.0113 (7)	0.0199 (7)	0.0147 (7)

Geometric parameters (Å, º)

O12—C12	1.2188 (17)	C31—C41	1.387 (2)
O21—C22	1.2180 (15)	C41—C51	1.375 (2)
O22—C22	1.3202 (15)	C51—C61	1.3926 (19)
O22—H22	0.94 (2)	C1—H1	0.9800
O211—C211	1.4255 (18)	C2—H2	0.9800
O211—C21	1.3705 (16)	C3—H3A	0.9700
N11—C11	1.4138 (16)	C3—H3B	0.9700
N11—C12	1.3520 (16)	C4—H4A	0.9700
N11—H11	0.867 (15)	C4—H4B	0.9700
C1—C12	1.5278 (17)	C5—H5A	0.9700
C1—C6	1.5355 (18)	C5—H5B	0.9700
C1—C2	1.5370 (16)	C6—H6A	0.9700
C2—C3	1.5413 (18)	C6—H6B	0.9700
C2—C22	1.5117 (17)	C31—H31	0.9300
C3—C4	1.5197 (19)	C41—H41	0.9300
C4—C5	1.5206 (18)	C51—H51	0.9300
C5—C6	1.5319 (19)	C61—H61	0.9300
C11—C61	1.3881 (19)	C211—H21A	0.9600
C11—C21	1.4021 (17)	C211—H21B	0.9600
C21—C31	1.3842 (18)	C211—H21C	0.9600
C22—O22—H22	106.5 (13)	C22—C2—H2	107.00

C21—O211—C211	117.55 (10)	C2—C3—H3A	109.00
C11—N11—C12	128.84 (12)	C2—C3—H3B	109.00
C12—N11—H11	119.8 (10)	C4—C3—H3A	109.00
C11—N11—H11	111.3 (10)	C4—C3—H3B	109.00
C2—C1—C6	112.86 (10)	H3A—C3—H3B	108.00
C2—C1—C12	109.56 (10)	C3—C4—H4A	109.00
C6—C1—C12	116.96 (10)	C3—C4—H4B	109.00
C1—C2—C3	110.70 (10)	C5—C4—H4A	109.00
C1—C2—C22	112.47 (10)	C5—C4—H4B	109.00
C3—C2—C22	111.18 (10)	H4A—C4—H4B	108.00
C2—C3—C4	111.44 (10)	C4—C5—H5A	109.00
C3—C4—C5	111.35 (11)	C4—C5—H5B	109.00
C4—C5—C6	112.20 (11)	C6—C5—H5A	109.00
C1—C6—C5	111.51 (11)	C6—C5—H5B	109.00
N11—C11—C21	116.06 (11)	H5A—C5—H5B	108.00
C21—C11—C61	119.37 (11)	C1—C6—H6A	109.00
N11—C11—C61	124.56 (11)	C1—C6—H6B	109.00
N11—C12—C1	116.37 (11)	C5—C6—H6A	109.00
O12—C12—C1	120.40 (11)	C5—C6—H6B	109.00
O12—C12—N11	123.19 (12)	H6A—C6—H6B	108.00
O211—C21—C11	114.89 (11)	C21—C31—H31	120.00
O211—C21—C31	124.91 (12)	C41—C31—H31	120.00
C11—C21—C31	120.20 (12)	C31—C41—H41	120.00
O22—C22—C2	112.97 (10)	C51—C41—H41	120.00
O21—C22—O22	122.14 (12)	C41—C51—H51	120.00
O21—C22—C2	124.89 (11)	C61—C51—H51	120.00
C21—C31—C41	119.62 (12)	C11—C61—H61	120.00
C31—C41—C51	120.77 (13)	C51—C61—H61	120.00
C41—C51—C61	119.94 (13)	O211—C211—H21A	110.00
C11—C61—C51	120.10 (12)	O211—C211—H21B	109.00
C2—C1—H1	106.00	O211—C211—H21C	109.00
C6—C1—H1	105.00	H21A—C211—H21B	109.00
C12—C1—H1	105.00	H21A—C211—H21C	109.00
C1—C2—H2	107.00	H21B—C211—H21C	109.00
C3—C2—H2	107.00		

C211—O211—C21—C11	173.23 (11)	C1—C2—C22—O21	−6.84 (17)
C211—O211—C21—C31	−7.53 (18)	C1—C2—C22—O22	173.17 (10)
C12—N11—C11—C21	175.18 (12)	C3—C2—C22—O21	117.95 (13)
C12—N11—C11—C61	−4.3 (2)	C3—C2—C22—O22	−62.04 (14)
C11—N11—C12—O12	1.7 (2)	C2—C3—C4—C5	−56.81 (14)
C11—N11—C12—C1	179.56 (11)	C3—C4—C5—C6	55.64 (15)
C6—C1—C2—C3	−52.76 (13)	C4—C5—C6—C1	−52.67 (14)
C6—C1—C2—C22	72.30 (13)	N11—C11—C21—O211	0.97 (16)
C12—C1—C2—C3	175.02 (10)	N11—C11—C21—C31	−178.31 (11)
C12—C1—C2—C22	−59.92 (13)	C61—C11—C21—O211	−179.48 (11)
C2—C1—C6—C5	51.65 (14)	C61—C11—C21—C31	1.24 (19)
C12—C1—C6—C5	−179.88 (10)	N11—C11—C61—C51	178.56 (12)
C2—C1—C12—O12	−31.79 (16)	C21—C11—C61—C51	−0.95 (19)

C2—C1—C12—N11	150.33 (11)	O211—C21—C31—C41	−179.81 (12)
C6—C1—C12—O12	−161.82 (12)	C11—C21—C31—C41	−0.61 (19)
C6—C1—C12—N11	20.30 (16)	C21—C31—C41—C51	−0.3 (2)
C1—C2—C3—C4	55.06 (13)	C31—C41—C51—C61	0.6 (2)
C22—C2—C3—C4	−70.72 (13)	C41—C51—C61—C11	0.0 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O211	0.867 (15)	2.119 (15)	2.6003 (14)	114.4 (12)
O22—H22···O21^{i}	0.94 (2)	1.76 (2)	2.6866 (14)	173 (2)
C3—H3*A*···O22	0.97	2.58	2.9294 (17)	101
C6—H6*B*···O21	0.97	2.56	3.1171 (16)	117
C51—H51···O12^{ii}	0.93	2.55	3.4232 (18)	157
C61—H61···O12	0.93	2.29	2.8812 (17)	121

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

(II) 2-(4-methoxyphenyl)-3a,4,5,6,7,7a-hexahydroisoindole-1,3-dione

Crystal data

$C_{15}H_{17}NO_3$
M_r = 259.30
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 11.7119 (5) Å
b = 6.6705 (3) Å
c = 17.2898 (8) Å
β = 109.482 (5)°
V = 1273.42 (11) Å^3
Z = 4
$F(000)$ = 552
D_x = 1.352 Mg m^{-3}
Melting point: 428 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4720 reflections
θ = 3.3–28.7°
μ = 0.09 mm^{-1}
T = 200 K
Flat prism, purple brown
0.40 × 0.25 × 0.12 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.929, T_{max} = 0.981
8552 measured reflections
2498 independent reflections
1959 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 26.0°, θ_{min} = 3.3°
h = −14→14
k = −8→8
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.088
S = 1.09
2498 reflections
172 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.60601 (9)	0.55074 (13)	0.38265 (6)	0.0389 (3)
O3	0.44297 (8)	0.03988 (12)	0.20453 (5)	0.0335 (3)
O41	0.69783 (9)	0.65459 (13)	0.03909 (6)	0.0371 (3)
N2	0.54233 (9)	0.31061 (14)	0.28078 (6)	0.0248 (3)
C1	0.55873 (11)	0.39094 (19)	0.35851 (8)	0.0274 (4)
C3	0.48336 (11)	0.12526 (17)	0.26984 (8)	0.0243 (4)
C4	0.38378 (12)	−0.08664 (18)	0.35410 (8)	0.0307 (4)
C5	0.26841 (12)	0.02011 (19)	0.35074 (9)	0.0337 (4)
C6	0.29309 (12)	0.18467 (19)	0.41557 (9)	0.0343 (5)
C7	0.37770 (12)	0.34107 (19)	0.40050 (8)	0.0316 (4)
C8	0.49925 (11)	0.25028 (18)	0.40214 (8)	0.0270 (4)
C9	0.48592 (12)	0.05478 (17)	0.35351 (7)	0.0263 (4)
C11	0.58123 (11)	0.40478 (18)	0.21881 (7)	0.0243 (4)
C21	0.54235 (11)	0.59554 (18)	0.19122 (8)	0.0270 (4)
C31	0.57910 (11)	0.68403 (18)	0.13073 (8)	0.0283 (4)
C41	0.65478 (11)	0.58023 (18)	0.09807 (8)	0.0266 (4)
C42	0.64175 (13)	0.8314 (2)	−0.00325 (9)	0.0400 (5)
C51	0.69393 (12)	0.38858 (18)	0.12627 (8)	0.0293 (4)
C61	0.65808 (11)	0.30128 (18)	0.18673 (8)	0.0272 (4)
H4A	0.36650	−0.17640	0.30750	0.0370*
H4B	0.41090	−0.16770	0.40350	0.0370*
H5A	0.21180	−0.07600	0.35960	0.0400*
H5B	0.23160	0.07860	0.29680	0.0400*
H6A	0.21740	0.24750	0.41350	0.0410*
H6B	0.32960	0.12670	0.46970	0.0410*
H7A	0.39280	0.44440	0.44220	0.0380*
H7B	0.33870	0.40360	0.34760	0.0380*
H8	0.55210	0.22960	0.45890	0.0320*
H9	0.56180	−0.01950	0.37740	0.0320*
H21	0.49140	0.66480	0.21320	0.0320*
H31	0.55300	0.81250	0.11220	0.0340*
H42A	0.65590	0.94100	0.03480	0.0480*
H42B	0.55620	0.80940	−0.02760	0.0480*
H42C	0.67550	0.86260	−0.04540	0.0480*
H51	0.74460	0.31890	0.10420	0.0350*
H61	0.68520	0.17370	0.20590	0.0330*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0453 (6)	0.0348 (5)	0.0367 (6)	−0.0172 (5)	0.0137 (5)	−0.0077 (4)
O3	0.0460 (6)	0.0277 (5)	0.0285 (5)	−0.0054 (4)	0.0147 (4)	−0.0053 (4)
O41	0.0447 (6)	0.0356 (5)	0.0411 (6)	0.0045 (4)	0.0279 (5)	0.0094 (4)
N2	0.0272 (6)	0.0238 (5)	0.0252 (6)	−0.0029 (4)	0.0112 (4)	0.0004 (4)
C1	0.0250 (7)	0.0285 (7)	0.0269 (7)	−0.0027 (5)	0.0063 (5)	−0.0008 (6)
C3	0.0247 (7)	0.0206 (6)	0.0289 (7)	0.0032 (5)	0.0105 (5)	0.0006 (5)
C4	0.0418 (8)	0.0227 (7)	0.0291 (7)	−0.0045 (6)	0.0138 (6)	0.0022 (6)
C5	0.0316 (7)	0.0332 (7)	0.0373 (8)	−0.0089 (6)	0.0127 (6)	0.0009 (6)
C6	0.0355 (8)	0.0366 (8)	0.0359 (8)	−0.0021 (6)	0.0187 (6)	0.0003 (6)
C7	0.0394 (8)	0.0256 (7)	0.0329 (7)	−0.0014 (6)	0.0161 (6)	−0.0032 (6)
C8	0.0307 (7)	0.0288 (7)	0.0200 (6)	−0.0048 (5)	0.0066 (5)	−0.0001 (5)
C9	0.0279 (7)	0.0238 (6)	0.0269 (7)	0.0024 (5)	0.0089 (5)	0.0046 (5)
C11	0.0237 (7)	0.0255 (6)	0.0246 (6)	−0.0025 (5)	0.0093 (5)	0.0004 (5)
C21	0.0253 (7)	0.0265 (7)	0.0326 (7)	0.0026 (5)	0.0143 (6)	−0.0007 (6)
C31	0.0296 (7)	0.0234 (6)	0.0331 (7)	0.0044 (5)	0.0122 (6)	0.0049 (6)
C41	0.0258 (7)	0.0282 (7)	0.0276 (7)	−0.0022 (5)	0.0112 (5)	0.0010 (5)
C42	0.0452 (9)	0.0434 (8)	0.0342 (8)	0.0015 (7)	0.0168 (7)	0.0125 (7)
C51	0.0272 (7)	0.0283 (7)	0.0372 (8)	0.0036 (5)	0.0171 (6)	−0.0005 (6)
C61	0.0244 (7)	0.0235 (6)	0.0339 (7)	0.0032 (5)	0.0100 (6)	0.0038 (5)

Geometric parameters (Å, º)

O1—C1	1.2095 (16)	C41—C51	1.3904 (17)
O3—C3	1.2112 (15)	C51—C61	1.3787 (19)
O41—C41	1.3723 (17)	C4—H4A	0.9700
O41—C42	1.4274 (17)	C4—H4B	0.9700
N2—C1	1.3993 (16)	C5—H5A	0.9700
N2—C3	1.3980 (15)	C5—H5B	0.9700
N2—C11	1.4408 (16)	C6—H6A	0.9700
C1—C8	1.5117 (18)	C6—H6B	0.9700
C3—C9	1.5119 (17)	C7—H7A	0.9700
C4—C5	1.511 (2)	C7—H7B	0.9700
C4—C9	1.5262 (19)	C8—H8	0.9800
C5—C6	1.5260 (19)	C9—H9	0.9800
C6—C7	1.520 (2)	C21—H21	0.9300
C7—C8	1.539 (2)	C31—H31	0.9300
C8—C9	1.5311 (17)	C42—H42A	0.9600
C11—C21	1.3817 (17)	C42—H42B	0.9600
C11—C61	1.3883 (18)	C42—H42C	0.9600
C21—C31	1.3884 (18)	C51—H51	0.9300
C31—C41	1.3848 (19)	C61—H61	0.9300
C41—O41—C42	117.36 (11)	C4—C5—H5A	109.00
C1—N2—C3	111.84 (10)	C4—C5—H5B	109.00
C1—N2—C11	124.83 (10)	C6—C5—H5A	109.00
C3—N2—C11	123.33 (10)	C6—C5—H5B	109.00
O1—C1—N2	124.70 (12)	H5A—C5—H5B	108.00

O1—C1—C8	127.51 (12)	C5—C6—H6A	110.00
N2—C1—C8	107.64 (10)	C5—C6—H6B	110.00
O3—C3—N2	124.32 (12)	C7—C6—H6A	110.00
O3—C3—C9	128.52 (11)	C7—C6—H6B	110.00
N2—C3—C9	107.12 (10)	H6A—C6—H6B	108.00
C5—C4—C9	113.66 (10)	C6—C7—H7A	109.00
C4—C5—C6	111.27 (12)	C6—C7—H7B	109.00
C5—C6—C7	110.02 (12)	C8—C7—H7A	109.00
C6—C7—C8	112.26 (11)	C8—C7—H7B	109.00
C1—C8—C7	108.91 (10)	H7A—C7—H7B	108.00
C1—C8—C9	103.54 (10)	C1—C8—H8	110.00
C7—C8—C9	113.63 (11)	C7—C8—H8	110.00
C3—C9—C4	115.67 (10)	C9—C8—H8	110.00
C3—C9—C8	103.09 (9)	C3—C9—H9	107.00
C4—C9—C8	117.35 (11)	C4—C9—H9	107.00
N2—C11—C21	120.58 (11)	C8—C9—H9	107.00
N2—C11—C61	119.23 (11)	C11—C21—H21	120.00
C21—C11—C61	120.20 (12)	C31—C21—H21	120.00
C11—C21—C31	120.15 (12)	C21—C31—H31	120.00
C21—C31—C41	119.73 (11)	C41—C31—H31	120.00
O41—C41—C31	124.48 (11)	O41—C42—H42A	109.00
O41—C41—C51	115.66 (12)	O41—C42—H42B	109.00
C31—C41—C51	119.85 (12)	O41—C42—H42C	109.00
C41—C51—C61	120.40 (13)	H42A—C42—H42B	109.00
C11—C61—C51	119.66 (11)	H42A—C42—H42C	110.00
C5—C4—H4A	109.00	H42B—C42—H42C	110.00
C5—C4—H4B	109.00	C41—C51—H51	120.00
C9—C4—H4A	109.00	C61—C51—H51	120.00
C9—C4—H4B	109.00	C11—C61—H61	120.00
H4A—C4—H4B	108.00	C51—C61—H61	120.00

C42—O41—C41—C31	−13.59 (19)	C5—C4—C9—C8	38.31 (15)
C42—O41—C41—C51	167.66 (12)	C5—C4—C9—C3	−83.79 (14)
C3—N2—C1—C8	−3.10 (14)	C9—C4—C5—C6	−50.57 (15)
C11—N2—C1—C8	177.11 (11)	C4—C5—C6—C7	61.34 (14)
C11—N2—C3—C9	166.12 (11)	C5—C6—C7—C8	−58.69 (15)
C1—N2—C11—C21	−56.72 (18)	C6—C7—C8—C1	160.25 (11)
C3—N2—C11—C21	123.52 (13)	C6—C7—C8—C9	45.42 (15)
C3—N2—C1—O1	−179.06 (13)	C7—C8—C9—C3	92.97 (13)
C11—N2—C1—O1	1.2 (2)	C7—C8—C9—C4	−35.41 (15)
C11—N2—C3—O3	−11.6 (2)	C1—C8—C9—C3	−25.01 (13)
C1—N2—C3—C9	−13.68 (14)	C1—C8—C9—C4	−153.39 (11)
C1—N2—C11—C61	123.53 (13)	N2—C11—C61—C51	178.75 (12)
C3—N2—C11—C61	−56.23 (17)	C21—C11—C61—C51	−1.00 (19)
C1—N2—C3—O3	168.63 (13)	N2—C11—C21—C31	−179.18 (12)
N2—C1—C8—C7	−103.15 (12)	C61—C11—C21—C31	0.57 (19)
O1—C1—C8—C9	−166.11 (14)	C11—C21—C31—C41	0.08 (19)
N2—C1—C8—C9	18.08 (13)	C21—C31—C41—C51	−0.3 (2)
O1—C1—C8—C7	72.67 (17)	C21—C31—C41—O41	−179.00 (12)

O3—C3—C9—C8	−158.30 (14)	O41—C41—C51—C61	178.67 (12)
N2—C3—C9—C4	153.56 (11)	C31—C41—C51—C61	−0.1 (2)
O3—C3—C9—C4	−28.9 (2)	C41—C51—C61—C11	0.8 (2)
N2—C3—C9—C8	24.14 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C21—H21···O3^{i}	0.93	2.56	3.2205 (15)	128
C51—H51···O1^{ii}	0.93	2.46	3.2914 (17)	149

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*x*+3/2, *y*−1/2, −*z*+1/2.

(III) *cis*-3-(1,3-dioxo-3a,4,5,6,7,7a-hexahydroisoindol-2-yl)benzoic acid

Crystal data

$C_{15}H_{15}NO_4$
M_r = 273.28
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 6.4958 (2) Å
b = 12.5236 (4) Å
c = 16.1281 (6) Å
V = 1312.03 (8) Å^3
Z = 4
$F(000)$ = 576
D_x = 1.383 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2474 reflections
θ = 3.3–28.7°
μ = 0.10 mm^{-1}
T = 200 K
Needles, colourless
0.45 × 0.12 × 0.08 mm

Data collection

Oxford Diffraction Gemini-S Ultra CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.980, T_{max} = 0.990
4693 measured reflections
1728 independent reflections
1395 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.0°, θ_{min} = 3.3°
h = −8→8
k = −16→12
l = −21→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.086
S = 1.10
1728 reflections
185 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.3742 (2)	0.03578 (12)	0.64532 (10)	0.0283 (5)
O3	−0.0620 (3)	0.20661 (15)	0.46989 (11)	0.0471 (7)
O31	0.3658 (3)	0.40076 (13)	0.77921 (11)	0.0336 (6)
O32	0.1156 (3)	0.41628 (12)	0.87409 (10)	0.0306 (5)
N2	0.1305 (3)	0.12973 (13)	0.57271 (11)	0.0190 (5)
C1	0.2977 (3)	0.06243 (16)	0.57948 (15)	0.0203 (7)
C3	0.0710 (4)	0.14497 (18)	0.49016 (15)	0.0267 (7)
C4	0.0431 (4)	−0.0268 (2)	0.41929 (17)	0.0337 (8)
C5	0.1575 (4)	−0.1282 (2)	0.39722 (17)	0.0394 (9)
C6	0.3037 (4)	−0.15842 (19)	0.46748 (18)	0.0407 (9)
C7	0.4642 (4)	−0.07326 (19)	0.47977 (17)	0.0352 (8)
C8	0.3732 (4)	0.03688 (18)	0.49230 (15)	0.0273 (7)
C9	0.1892 (4)	0.06670 (18)	0.43767 (15)	0.0281 (8)
C11	0.0356 (3)	0.18358 (16)	0.64192 (14)	0.0176 (6)
C21	0.1500 (3)	0.25567 (16)	0.68784 (13)	0.0190 (6)
C31	0.0592 (3)	0.30532 (17)	0.75634 (13)	0.0201 (6)
C41	−0.1440 (3)	0.28329 (18)	0.77614 (15)	0.0248 (7)
C51	−0.2570 (3)	0.21244 (19)	0.72850 (15)	0.0280 (7)
C61	−0.1674 (3)	0.16229 (17)	0.66081 (15)	0.0235 (7)
C311	0.1794 (3)	0.37990 (17)	0.80984 (15)	0.0216 (7)
H4A	−0.04230	−0.03990	0.46760	0.0400*
H4B	−0.04680	−0.00740	0.37370	0.0400*
H5A	0.23510	−0.11750	0.34650	0.0470*
H5B	0.06000	−0.18560	0.38800	0.0470*
H6A	0.22610	−0.16770	0.51830	0.0490*
H6B	0.37050	−0.22570	0.45450	0.0490*
H7A	0.55400	−0.07180	0.43170	0.0420*
H7B	0.54740	−0.09140	0.52770	0.0420*
H8	0.48270	0.08820	0.47970	0.0330*
H9	0.23550	0.10040	0.38610	0.0340*
H21	0.28550	0.27090	0.67330	0.0230*
H31	0.427 (4)	0.444 (2)	0.8157 (19)	0.049 (9)*
H41	−0.20480	0.31630	0.82170	0.0300*
H51	−0.39360	0.19840	0.74190	0.0340*
H61	−0.24330	0.11490	0.62860	0.0280*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0247 (9)	0.0310 (8)	0.0292 (9)	0.0063 (8)	−0.0091 (8)	0.0012 (7)
O3	0.0641 (13)	0.0478 (11)	0.0293 (10)	0.0328 (11)	−0.0152 (10)	−0.0065 (9)
O31	0.0260 (9)	0.0416 (10)	0.0332 (10)	−0.0113 (9)	0.0027 (8)	−0.0162 (8)

O32	0.0373 (10)	0.0321 (8)	0.0225 (9)	−0.0037 (8)	0.0027 (8)	−0.0083 (8)
N2	0.0194 (9)	0.0179 (8)	0.0197 (10)	0.0011 (8)	−0.0033 (8)	−0.0012 (7)
C1	0.0166 (11)	0.0141 (10)	0.0303 (13)	−0.0029 (9)	−0.0039 (10)	−0.0014 (10)
C3	0.0352 (14)	0.0235 (11)	0.0215 (12)	0.0037 (11)	−0.0058 (11)	−0.0011 (10)
C4	0.0342 (14)	0.0375 (14)	0.0294 (14)	0.0048 (13)	−0.0093 (13)	−0.0062 (12)
C5	0.0484 (17)	0.0354 (13)	0.0344 (15)	0.0015 (14)	−0.0098 (14)	−0.0127 (12)
C6	0.0509 (18)	0.0283 (13)	0.0429 (17)	0.0087 (13)	−0.0094 (15)	−0.0073 (12)
C7	0.0299 (14)	0.0391 (14)	0.0367 (15)	0.0108 (13)	−0.0038 (12)	−0.0102 (12)
C8	0.0242 (12)	0.0299 (12)	0.0279 (13)	−0.0015 (11)	0.0001 (12)	−0.0064 (10)
C9	0.0358 (14)	0.0271 (12)	0.0215 (13)	0.0061 (11)	0.0007 (11)	−0.0008 (10)
C11	0.0181 (11)	0.0158 (10)	0.0188 (11)	0.0030 (9)	−0.0038 (10)	0.0007 (9)
C21	0.0169 (11)	0.0203 (10)	0.0198 (11)	−0.0011 (10)	−0.0014 (10)	0.0013 (9)
C31	0.0216 (11)	0.0198 (10)	0.0188 (11)	0.0009 (10)	−0.0023 (10)	0.0024 (9)
C41	0.0229 (12)	0.0293 (11)	0.0223 (11)	0.0034 (11)	0.0035 (11)	−0.0024 (10)
C51	0.0169 (11)	0.0346 (13)	0.0326 (14)	−0.0040 (11)	0.0019 (11)	0.0029 (12)
C61	0.0194 (12)	0.0218 (11)	0.0292 (13)	−0.0035 (10)	−0.0051 (10)	−0.0010 (10)
C311	0.0223 (12)	0.0202 (11)	0.0223 (12)	0.0026 (10)	−0.0018 (10)	0.0016 (9)

Geometric parameters (Å, º)

O1—C1	1.219 (3)	C31—C41	1.386 (3)
O3—C3	1.204 (3)	C31—C311	1.492 (3)
O31—C311	1.334 (3)	C41—C51	1.384 (3)
O32—C311	1.206 (3)	C51—C61	1.388 (3)
O31—H31	0.89 (3)	C4—H4A	0.9700
N2—C3	1.399 (3)	C4—H4B	0.9700
N2—C11	1.443 (3)	C5—H5A	0.9700
N2—C1	1.379 (3)	C5—H5B	0.9700
C1—C8	1.523 (3)	C6—H6A	0.9700
C3—C9	1.506 (3)	C6—H6B	0.9700
C4—C9	1.536 (4)	C7—H7A	0.9700
C4—C5	1.514 (4)	C7—H7B	0.9700
C5—C6	1.526 (4)	C8—H8	0.9800
C6—C7	1.505 (4)	C9—H9	0.9800
C7—C8	1.514 (3)	C21—H21	0.9300
C8—C9	1.531 (4)	C41—H41	0.9300
C11—C61	1.379 (3)	C51—H51	0.9300
C11—C21	1.384 (3)	C61—H61	0.9300
C21—C31	1.398 (3)		
C311—O31—H31	106.2 (18)	C9—C4—H4A	109.00
C1—N2—C3	112.10 (19)	C4—C5—H5A	110.00
C3—N2—C11	123.67 (18)	C4—C5—H5B	110.00
C1—N2—C11	124.13 (18)	C6—C5—H5A	110.00
O1—C1—C8	127.98 (19)	C6—C5—H5B	110.00
N2—C1—C8	107.99 (19)	H5A—C5—H5B	108.00
O1—C1—N2	123.9 (2)	C7—C6—H6A	110.00
O3—C3—N2	123.0 (2)	C7—C6—H6B	110.00
N2—C3—C9	107.78 (19)	C5—C6—H6B	109.00
O3—C3—C9	129.1 (2)	C5—C6—H6A	110.00

C5—C4—C9	112.4 (2)	H6A—C6—H6B	108.00
C4—C5—C6	109.8 (2)	H7A—C7—H7B	108.00
C5—C6—C7	110.7 (2)	C6—C7—H7B	109.00
C6—C7—C8	113.1 (2)	C8—C7—H7B	109.00
C1—C8—C7	116.1 (2)	C6—C7—H7A	109.00
C7—C8—C9	116.8 (2)	C8—C7—H7A	109.00
C1—C8—C9	103.22 (19)	C1—C8—H8	107.00
C3—C9—C4	106.8 (2)	C7—C8—H8	107.00
C4—C9—C8	114.04 (19)	C9—C8—H8	107.00
C3—C9—C8	103.49 (19)	C3—C9—H9	111.00
N2—C11—C61	119.28 (19)	C8—C9—H9	111.00
C21—C11—C61	121.4 (2)	C4—C9—H9	111.00
N2—C11—C21	119.31 (18)	C11—C21—H21	120.00
C11—C21—C31	119.10 (18)	C31—C21—H21	120.00
C21—C31—C41	119.69 (19)	C51—C41—H41	120.00
C21—C31—C311	120.97 (18)	C31—C41—H41	120.00
C41—C31—C311	119.33 (19)	C41—C51—H51	120.00
C31—C41—C51	120.3 (2)	C61—C51—H51	120.00
C41—C51—C61	120.29 (19)	C11—C61—H61	120.00
C11—C61—C51	119.2 (2)	C51—C61—H61	120.00
H4A—C4—H4B	108.00	O31—C311—C31	112.55 (19)
C5—C4—H4B	109.00	O32—C311—C31	123.63 (19)
C9—C4—H4B	109.00	O31—C311—O32	123.8 (2)
C5—C4—H4A	109.00		

C3—N2—C1—O1	−178.5 (2)	C4—C5—C6—C7	−62.6 (3)
C3—N2—C1—C8	5.7 (2)	C5—C6—C7—C8	53.8 (3)
C11—N2—C1—O1	5.1 (3)	C6—C7—C8—C1	81.3 (3)
C11—N2—C1—C8	−170.65 (18)	C6—C7—C8—C9	−40.9 (3)
C1—N2—C3—O3	−174.6 (2)	C1—C8—C9—C3	22.7 (2)
C1—N2—C3—C9	9.7 (3)	C1—C8—C9—C4	−93.0 (2)
C11—N2—C3—O3	1.8 (4)	C7—C8—C9—C3	151.4 (2)
C11—N2—C3—C9	−173.96 (19)	C7—C8—C9—C4	35.7 (3)
C1—N2—C11—C21	61.8 (3)	N2—C11—C21—C31	−178.14 (18)
C1—N2—C11—C61	−118.4 (2)	C61—C11—C21—C31	2.0 (3)
C3—N2—C11—C21	−114.2 (2)	N2—C11—C61—C51	178.6 (2)
C3—N2—C11—C61	65.7 (3)	C21—C11—C61—C51	−1.5 (3)
O1—C1—C8—C7	37.3 (3)	C11—C21—C31—C41	−1.2 (3)
O1—C1—C8—C9	166.3 (2)	C11—C21—C31—C311	177.28 (19)
N2—C1—C8—C7	−147.2 (2)	C21—C31—C41—C51	0.1 (3)
N2—C1—C8—C9	−18.1 (2)	C311—C31—C41—C51	−178.5 (2)
O3—C3—C9—C4	−75.2 (3)	C21—C31—C311—O31	7.1 (3)
O3—C3—C9—C8	164.1 (3)	C21—C31—C311—O32	−172.2 (2)
N2—C3—C9—C4	100.2 (2)	C41—C31—C311—O31	−174.4 (2)
N2—C3—C9—C8	−20.5 (2)	C41—C31—C311—O32	6.3 (3)
C9—C4—C5—C6	57.4 (3)	C31—C41—C51—C61	0.4 (3)
C5—C4—C9—C3	−157.6 (2)	C41—C51—C61—C11	0.3 (3)
C5—C4—C9—C8	−43.9 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O31—H31···O1^{i}	0.89 (3)	1.84 (3)	2.682 (2)	156 (3)
C21—H21···O3^{ii}	0.93	2.53	3.193 (3)	129

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+3/2; (ii) *x*+1/2, −*y*+1/2, −*z*+1.

(IV) *rac*-*cis*-4-(1,3-dioxo-3a,4,5,6,7,7a-hexahydroisoindol- 2-yl)benzoic acid monohydrate

Crystal data

$C_{15}H_{15}NO_4·H_2O$
M_r = 291.30
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 13.077 (2) Å
b = 6.6713 (7) Å
c = 16.432 (2) Å
β = 97.982 (13)°
V = 1419.6 (3) Å^3
Z = 4
$F(000)$ = 616
D_x = 1.363 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2417 reflections
θ = 3.1–28.7°
μ = 0.10 mm^{-1}
T = 200 K
Prism, colourless
0.30 × 0.10 × 0.08 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.813, T_{max} = 0.980
9649 measured reflections
2790 independent reflections
1374 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 26.0°, θ_{min} = 3.2°
h = −16→13
k = −8→8
l = −14→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.132
S = 0.85
2790 reflections
194 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0704P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.80344 (15)	0.0890 (3)	0.54274 (11)	0.0502 (7)	
O3	0.58298 (15)	0.6120 (2)	0.56137 (11)	0.0480 (7)	
O41	0.50408 (17)	−0.2147 (3)	0.82985 (12)	0.0632 (9)	
O42	0.53908 (18)	0.0528 (3)	0.90848 (13)	0.0732 (10)	
N2	0.68963 (16)	0.3350 (3)	0.57093 (12)	0.0333 (7)	
C1	0.7620 (2)	0.2489 (4)	0.52623 (16)	0.0370 (9)	
C3	0.6525 (2)	0.5193 (3)	0.53794 (15)	0.0371 (10)	
C4	0.6735 (3)	0.7105 (4)	0.40365 (19)	0.0664 (14)	
C5	0.6294 (3)	0.5965 (5)	0.3295 (2)	0.0707 (14)	
C6	0.7017 (3)	0.4390 (4)	0.30655 (18)	0.0656 (13)	
C7	0.7239 (3)	0.2867 (4)	0.37592 (16)	0.0580 (13)	
C8	0.7723 (2)	0.3861 (4)	0.45460 (15)	0.0422 (10)	
C9	0.7219 (2)	0.5812 (4)	0.47533 (17)	0.0490 (10)	
C11	0.65230 (19)	0.2395 (3)	0.64028 (14)	0.0312 (8)	
C21	0.6115 (2)	0.0499 (3)	0.63172 (15)	0.0361 (9)	
C31	0.5750 (2)	−0.0399 (4)	0.69778 (15)	0.0376 (9)	
C41	0.57975 (19)	0.0608 (4)	0.77213 (15)	0.0341 (9)	
C51	0.6220 (2)	0.2516 (4)	0.78004 (16)	0.0397 (9)	
C61	0.6595 (2)	0.3428 (4)	0.71395 (15)	0.0370 (9)	
C411	0.5398 (2)	−0.0315 (4)	0.84418 (18)	0.0461 (11)	
O1W	0.9383 (2)	−0.1718 (3)	0.46543 (15)	0.0644 (10)	0.810
O2W	0.9381 (10)	0.0061 (18)	0.4139 (7)	0.079 (3)*	0.190
H4A	0.61920	0.79100	0.42180	0.0800*	
H4B	0.72560	0.80150	0.38870	0.0800*	
H5A	0.61330	0.68870	0.28380	0.0850*	
H5B	0.56550	0.53340	0.33950	0.0850*	
H6A	0.76580	0.50090	0.29610	0.0790*	
H6B	0.67120	0.37170	0.25670	0.0790*	
H7A	0.66000	0.22200	0.38490	0.0690*	
H7B	0.77020	0.18440	0.36030	0.0690*	
H8	0.84560	0.41010	0.45180	0.0510*	
H9	0.77710	0.66240	0.50550	0.0590*	
H21	0.60850	−0.01750	0.58190	0.0430*	
H31	0.54720	−0.16830	0.69240	0.0450*	
H41	0.48190	−0.25400	0.87690	0.0950*	
H51	0.62530	0.31920	0.82980	0.0480*	
H61	0.68860	0.47010	0.71910	0.0440*	
H11W	0.89650	−0.09080	0.47990	0.0970*	0.810
H12W	0.94650	−0.26180	0.49990	0.0970*	0.810
H21W	0.89720	0.02910	0.45270	0.1180*	0.190
H22W	0.96620	0.12910	0.41170	0.1180*	0.190

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0626 (14)	0.0463 (11)	0.0474 (12)	0.0216 (10)	0.0273 (10)	0.0153 (9)

O3	0.0564 (14)	0.0348 (10)	0.0570 (13)	0.0078 (9)	0.0228 (11)	−0.0050 (9)
O41	0.0800 (17)	0.0664 (14)	0.0488 (13)	−0.0164 (11)	0.0285 (12)	0.0104 (10)
O42	0.0885 (19)	0.1014 (17)	0.0363 (13)	−0.0290 (13)	0.0319 (12)	−0.0105 (12)
N2	0.0435 (15)	0.0305 (11)	0.0289 (12)	0.0023 (9)	0.0157 (11)	−0.0005 (9)
C1	0.0432 (18)	0.0394 (15)	0.0311 (15)	0.0026 (13)	0.0148 (13)	0.0002 (12)
C3	0.0484 (19)	0.0287 (14)	0.0355 (16)	−0.0019 (12)	0.0102 (14)	−0.0047 (11)
C4	0.091 (3)	0.0535 (19)	0.059 (2)	0.0228 (17)	0.026 (2)	0.0202 (16)
C5	0.070 (3)	0.071 (2)	0.065 (2)	0.0037 (18)	−0.012 (2)	0.0190 (18)
C6	0.093 (3)	0.063 (2)	0.0380 (18)	0.0043 (18)	−0.0004 (18)	0.0000 (15)
C7	0.090 (3)	0.0442 (17)	0.0397 (18)	−0.0029 (15)	0.0088 (18)	−0.0030 (13)
C8	0.054 (2)	0.0395 (15)	0.0356 (16)	0.0060 (12)	0.0150 (14)	0.0066 (12)
C9	0.066 (2)	0.0412 (16)	0.0426 (17)	0.0025 (14)	0.0178 (15)	0.0070 (13)
C11	0.0349 (16)	0.0317 (13)	0.0291 (14)	0.0027 (11)	0.0120 (12)	−0.0015 (11)
C21	0.0458 (18)	0.0348 (15)	0.0299 (15)	0.0016 (12)	0.0133 (13)	−0.0035 (11)
C31	0.0413 (18)	0.0350 (14)	0.0378 (16)	−0.0031 (11)	0.0102 (13)	−0.0014 (12)
C41	0.0306 (16)	0.0423 (15)	0.0312 (15)	0.0007 (11)	0.0107 (12)	0.0009 (12)
C51	0.0389 (17)	0.0520 (16)	0.0298 (15)	−0.0018 (13)	0.0104 (13)	−0.0099 (12)
C61	0.0335 (16)	0.0390 (14)	0.0402 (16)	−0.0046 (11)	0.0113 (13)	−0.0078 (13)
C411	0.0396 (18)	0.061 (2)	0.0399 (18)	−0.0030 (14)	0.0136 (14)	0.0037 (15)
O1W	0.078 (2)	0.0581 (14)	0.0693 (18)	0.0183 (13)	0.0533 (15)	0.0055 (13)

Geometric parameters (Å, º)

O1—C1	1.210 (3)	C11—C61	1.385 (3)
O3—C3	1.206 (3)	C21—C31	1.382 (3)
O41—C411	1.318 (3)	C31—C41	1.388 (4)
O42—C411	1.198 (4)	C41—C411	1.492 (4)
O41—H41	0.9000	C41—C51	1.387 (4)
O1W—H12W	0.8200	C51—C61	1.392 (4)
O1W—H11W	0.8300	C4—H4B	0.9700
O2W—H22W	0.9000	C4—H4A	0.9700
O2W—H21W	0.9000	C5—H5B	0.9700
N2—C1	1.399 (3)	C5—H5A	0.9700
N2—C3	1.403 (3)	C6—H6B	0.9700
N2—C11	1.448 (3)	C6—H6A	0.9700
C1—C8	1.512 (4)	C7—H7A	0.9700
C3—C9	1.521 (4)	C7—H7B	0.9700
C4—C5	1.483 (5)	C8—H8	0.9800
C4—C9	1.525 (4)	C9—H9	0.9800
C5—C6	1.496 (5)	C21—H21	0.9300
C6—C7	1.525 (4)	C31—H31	0.9300
C7—C8	1.512 (4)	C51—H51	0.9300
C8—C9	1.519 (4)	C61—H61	0.9300
C11—C21	1.372 (3)		

C411—O41—H41	105.00	C9—C4—H4A	109.00
H11W—O1W—H12W	108.00	C9—C4—H4B	109.00
H21W—O2W—H22W	99.00	C4—C5—H5B	109.00
C1—N2—C3	112.3 (2)	C6—C5—H5A	109.00
C1—N2—C11	124.0 (2)	H5A—C5—H5B	108.00

C3—N2—C11	123.6 (2)	C4—C5—H5A	109.00
N2—C1—C8	107.5 (2)	C6—C5—H5B	109.00
O1—C1—C8	128.5 (2)	C5—C6—H6B	110.00
O1—C1—N2	124.0 (2)	C7—C6—H6A	110.00
N2—C3—C9	107.0 (2)	H6A—C6—H6B	108.00
O3—C3—N2	124.2 (2)	C7—C6—H6B	110.00
O3—C3—C9	128.6 (2)	C5—C6—H6A	110.00
C5—C4—C9	114.6 (2)	C8—C7—H7B	109.00
C4—C5—C6	112.3 (3)	C6—C7—H7A	109.00
C5—C6—C7	109.9 (3)	C6—C7—H7B	109.00
C6—C7—C8	111.2 (2)	C8—C7—H7A	109.00
C1—C8—C7	109.2 (2)	H7A—C7—H7B	108.00
C7—C8—C9	115.1 (2)	C7—C8—H8	109.00
C1—C8—C9	104.8 (2)	C9—C8—H8	109.00
C3—C9—C4	117.0 (2)	C1—C8—H8	109.00
C4—C9—C8	117.3 (2)	C8—C9—H9	106.00
C3—C9—C8	103.7 (2)	C3—C9—H9	106.00
N2—C11—C61	118.7 (2)	C4—C9—H9	106.00
C21—C11—C61	121.8 (2)	C11—C21—H21	120.00
N2—C11—C21	119.6 (2)	C31—C21—H21	120.00
C11—C21—C31	119.4 (2)	C21—C31—H31	120.00
C21—C31—C41	120.2 (2)	C41—C31—H31	120.00
C31—C41—C51	119.7 (2)	C41—C51—H51	120.00
C51—C41—C411	118.9 (2)	C61—C51—H51	120.00
C31—C41—C411	121.4 (2)	C11—C61—H61	121.00
C41—C51—C61	120.5 (2)	C51—C61—H61	121.00
C11—C61—C51	118.4 (2)	O42—C411—C41	123.6 (2)
H4A—C4—H4B	108.00	O41—C411—O42	123.2 (3)
C5—C4—H4A	109.00	O41—C411—C41	113.3 (2)
C5—C4—H4B	109.00		

C3—N2—C1—O1	−179.1 (2)	C4—C5—C6—C7	61.6 (4)
C3—N2—C1—C8	−1.8 (3)	C5—C6—C7—C8	−59.7 (4)
C11—N2—C1—O1	−2.7 (4)	C6—C7—C8—C1	162.2 (3)
C11—N2—C1—C8	174.6 (2)	C6—C7—C8—C9	44.7 (4)
C1—N2—C3—O3	172.9 (2)	C1—C8—C9—C3	−20.8 (3)
C1—N2—C3—C9	−11.9 (3)	C1—C8—C9—C4	−151.4 (2)
C11—N2—C3—O3	−3.5 (4)	C7—C8—C9—C3	99.2 (3)
C11—N2—C3—C9	171.7 (2)	C7—C8—C9—C4	−31.4 (4)
C1—N2—C11—C21	−53.8 (3)	N2—C11—C21—C31	−179.3 (2)
C1—N2—C11—C61	125.9 (3)	C61—C11—C21—C31	1.0 (4)
C3—N2—C11—C21	122.2 (3)	N2—C11—C61—C51	179.0 (2)
C3—N2—C11—C61	−58.2 (3)	C21—C11—C61—C51	−1.4 (4)
O1—C1—C8—C7	67.9 (4)	C11—C21—C31—C41	−0.1 (4)
O1—C1—C8—C9	−168.3 (3)	C21—C31—C41—C51	−0.5 (4)
N2—C1—C8—C7	−109.3 (3)	C21—C31—C41—C411	179.3 (2)
N2—C1—C8—C9	14.6 (3)	C31—C41—C51—C61	0.1 (4)
O3—C3—C9—C4	−34.1 (4)	C411—C41—C51—C61	−179.6 (2)
O3—C3—C9—C8	−164.9 (3)	C31—C41—C411—O41	2.0 (4)

N2—C3—C9—C4	151.1 (2)	C31—C41—C411—O42	−177.3 (3)
N2—C3—C9—C8	20.3 (3)	C51—C41—C411—O41	−178.3 (2)
C9—C4—C5—C6	−47.3 (4)	C51—C41—C411—O42	2.4 (4)
C5—C4—C9—C3	−91.9 (3)	C41—C51—C61—C11	0.8 (4)
C5—C4—C9—C8	32.3 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O41—H41···O1*W*^{i}	0.90	1.71	2.609 (3)	179
O1*W*—H11*W*···O1	0.83	2.08	2.894 (3)	167
O1*W*—H12*W*···O42^{ii}	0.82	1.94	2.754 (3)	172
O2*W*—H21*W*···O1	0.90	2.10	2.991 (12)	179
O2*W*—H22*W*···O42^{iii}	0.90	2.33	3.233 (12)	178
C9—H9···O1*W*^{iv}	0.98	2.55	3.298 (4)	133
C21—H21···O3^{v}	0.93	2.51	3.145 (3)	126
C31—H31···O41	0.93	2.42	2.737 (3)	100

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