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Intermolecular O—H...O hydrogen bonding in the three independent molecules of (2*S*)-3-(4-hydroxyphenyl)-2-(1-oxoisoindolin-2-yl)propanoic acid

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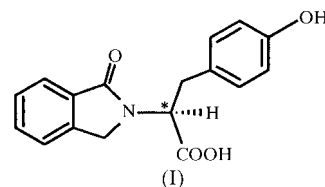
The title compound, $C_{17}H_{15}NO_4$, derived from L-tyrosine, crystallizes with three independent molecules which differ in the conformation of the asymmetric unit: the N—C—C— C_{ipso} torsion angles are $-71.7(5)$, $-63.6(6)$ and $-52.5(5)^\circ$, respectively. Deformations in the phenol ring hydroxy O—C—C angles of $116.5(4)/123.9(4)$, $121.7(5)/118.1(4)$ and $122.4(5)/118.6(5)^\circ$, respectively, result from their respective intermolecular hydrogen-bonding environments. Intermolecular $O_{acid}-H \cdots O=C_{indole}$, $O_{phenol}-H \cdots O-H_{phenol}$ and $O_{phenol}-H \cdots O=C_{indole}$ hydrogen bonds, with O...O distances in the range $2.607(4)$ – $2.809(4)$ Å, are present in combination with C—H...O and C—H... π_{arene} interactions. The primary hydrogen-bonding systems assemble with graph sets $R_3^3(8)$ and $R_3^2(22)$.

Comment

Phenol and related monosubstituted aromatic compounds have attracted considerable interest in gas-phase electron diffraction experiments and *ab initio* calculations in order to probe bond-angle deformations arising from electronic effects due to the nature of the phenyl ring substituent (Portalone *et al.*, 1992; Ramondo *et al.*, 1995). These investigations facilitate comparison with microwave and solid-state studies, although the experimental geometries in the 'free' molecule and crystal structure usually differ due to solid-state intra- and intermolecular interactions. Phenol crystallizes with three independent molecules in the asymmetric unit and intermolecular O—H...O hydrogen bonds form a helical array of approximate 3_1 symmetry (Zavodnik *et al.*, 1987). Many hydrocarbons containing a single OH substituent, *e.g.* menthol (Bombicz *et al.*, 1999) and 5α -dinosteran-29-ol (Brock *et al.*, 1994), crystallize with more than one molecule in the asymmetric unit or in space groups with three- or fourfold axes in the absence of other significant interactions.

Amino acid derivatives are a major class of chiral compounds with a wide range of applications in medicinal

chemistry. Tyrosine, $H_2N(H)C^*(R)COOH$ (*R* is 4-hydroxybenzyl) and related compounds (Byrkjedal *et al.*, 1974; Dahaoui *et al.*, 1999) have attracted considerable interest in biological studies (Kawai *et al.*, 1999). The title compound, (I), which contains the isoindolinyl group (Allin *et al.*, 1996; McNab *et al.*, 1997; Takahashi & Hatanaka, 1997) is synthesized from L-tyrosine and forms part of a study of the hydrogen-bonding interactions in a phthalimidine series (Brady *et al.*, 1998; Gallagher & Murphy, 1999; Gallagher & Brady, 2000; Gallagher *et al.*, 2000).



Compound (I) crystallizes in space group $P2_12_12_1$ (No. 19) with three independent molecules, *A*, *B* and *C*, in the asymmetric unit which differ in conformation but retain the same configuration (*S*) at the (H) $C^*(R)COOH$ chiral centre. The absolute structure can be deduced from the known absolute configuration of L-tyrosine used in the synthesis. Views of the three independent molecules, with the atomic numbering schemes, are given in Fig. 1 and selected dimensions are in Table 1.

The r.m.s. deviations for the superposition of the non-H atoms of any pair of molecules in (I) using *PLATON* (Spek, 1998) are 0.28 (*A/B* pair), 1.05 (*A/C* pair) and 0.56 Å (*B/C* pair). Crystal structures with several molecules present in the asymmetric unit are relatively common (Gallagher *et al.*, 1998). There are *ca* 45 molecules in the Cambridge Structural Database (Allen & Kennard, 1993) with $Z = 12$ or three molecules in the asymmetric unit in space group $P2_12_12_1$, of which *N*-acetyl-L-leucine-L-tyrosine methyl ester is a relevant example (Karle & Flippen-Anderson, 1989).

The bond lengths and angles in the five-membered rings of the three independent molecules of (I) are similar to those reported previously (McNab *et al.*, 1997; Kundu *et al.*, 1999; Mukherjee *et al.*, 2000; Gallagher & Brady, 2000) and in agreement with expected values (Orpen *et al.*, 1994). The angles between the five- and six-membered rings of the isoindole systems are $0.96(12)$, $2.87(8)$ and $1.01(7)^\circ$ in molecules *A*, *B* and *C*, respectively, and the maximum deviation from planarity for an atom in either ring plane is $0.021(3)$ Å for N1*A* in *A*, $0.021(3)$ Å for C3*B* in *B* and $0.015(3)$ Å for C10*C* in *C*. The carbonyl O3*n* atom is $0.067(7)$ Å from the C_4N ring plane in *A*, $0.113(7)$ Å in *B* and $0.020(7)$ Å in *C* (*n* is *A*, *B* or *C*). Pyramidalization occurs, with N1*n* $0.118(4)$, $0.121(5)$ and $0.070(5)$ Å from the $C2n/C3n/C10n$ planes, and these values are comparable with those of $0.129(2)$ Å in a DL-phenylalanine derivative (Brady *et al.*, 1998) and $0.079(2)$ Å in a DL-*meta*-tyrosine derivative (Gallagher & Murphy, 1999). The carboxylic acid group CCO_2 is almost perpendicular to the C_4N ring plane [$79.18(18)$, $83.3(2)$ and $83.0(2)^\circ$ in *A*, *B* and *C*, respectively] and the phenyl ring [$79.14(13)$, $68.39(14)$ and $82.5(2)^\circ$ in *A*, *B* and *C*, respectively]. Geometrical

differences for $O2n-C1n-C2n-C21n$, with torsion angles of 132.3 (5), 113.0 (6) and 128.3 (6) $^\circ$, respectively, are due to the distinct local environments of *A*, *B* and *C*.

Intermolecular hydrogen bonding involving the phenol residues can be evidenced by analysis of the $O4n-C14n-$

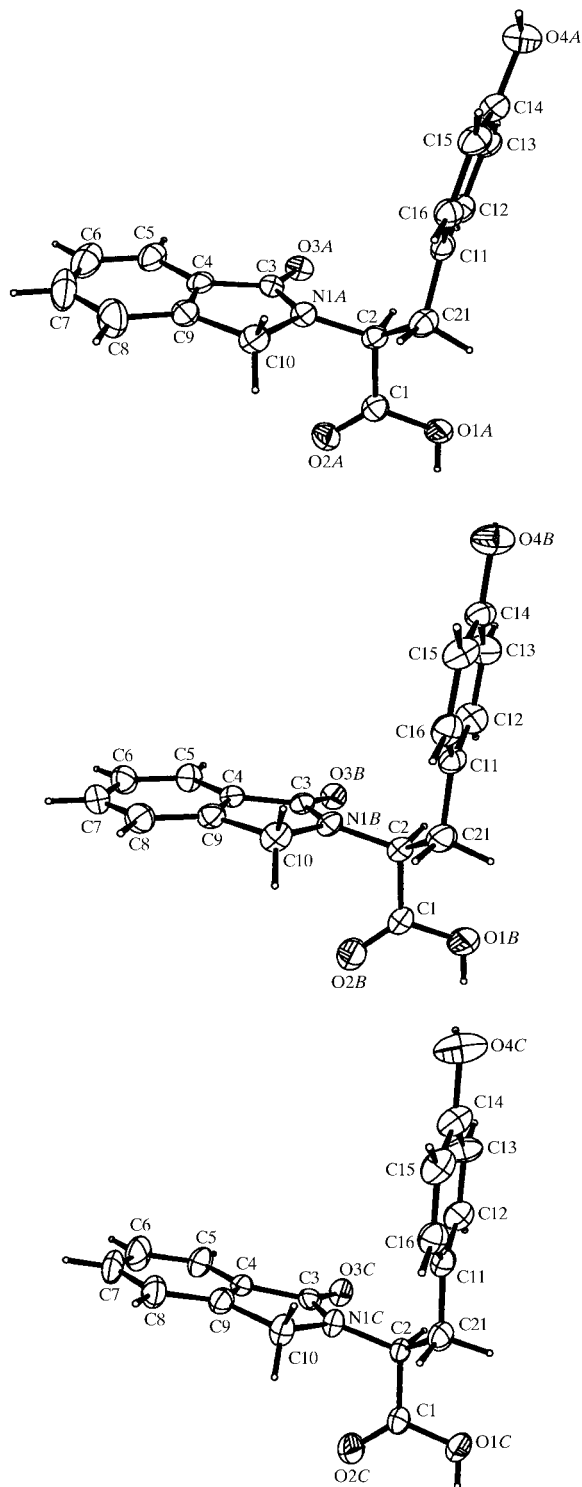


Figure 1
Views of molecules *A* (top), *B* and *C* (bottom) of (I) with the atomic numbering schemes. Displacement ellipsoids are drawn at the 30% probability level. Suffixes have been omitted from the C-atom labels for clarity.

$C13n/O4n-C14n-C15n$ bond angles, which vary as 116.5 (4)/ 123.9 (4), 121.7 (5)/ 118.1 (4) and 122.4 (5)/ 118.6 (5) $^\circ$ in *A*, *B* and *C*, respectively, with the larger angles *cisoid* to their O—H bonds, reflecting the different demands of O—H \cdots O hydrogen bonding at each O—H site (the $H4n-O4n-C14n-C13n$ angles are 169 , 8 and 20 $^\circ$). This deformation is also present in the *meta*-tyrosine derivative, where a 5 $^\circ$ difference in the O—C—*C*_{ortho} angles [117.2 (2)/ 122.2 (2) $^\circ$] results from strong intermolecular hydrogen bonding [the C—C—C angle is 118.9 (2) $^\circ$; Gallagher & Murphy, 1999]. In related alkyl —OH derivatives, the donor/acceptor ability of the alkyl —OH group in O—H \cdots O intermolecular hydrogen bonding can be noted from the O—C—C angle in the chiral and racemic threonine forms [110.5 (2) and 105.52 (11) $^\circ$, respectively; Gallagher *et al.*, 2000]. In tris(4-methoxyphenyl)methanol, MeO—C—*C*_{ortho} angles of 115.1 (3)/ 125.5 (3) $^\circ$ *transoid/cisoid* to a methoxy group are distinct (Ferguson *et al.*, 1996) and similar in magnitude to the values of $116/124$ $^\circ$ reported in the electron diffraction study of anisole, C₆H₅OCH₃ (Seip & Seip, 1973).

The hydrogen bonding in (I) consists primarily of O—H \cdots O interactions, with some C—H \cdots O and C—H $\cdots\pi$ _{arene} interactions, as detailed in Table 2. The intermolecular interactions are dominated by the O—H \cdots O hydrogen bonds with all six O—H groups participating; O \cdots O distances range from 2.607 (4) (O4*B* \cdots O3*C*) to 2.809 (4) Å (O4*C* \cdots O3*B*). The crystal structure can be considered as a molecular association of *B* and *C* through two O_{phenol}—H \cdots O=C_{indole} hydrogen bonds to form a dimer, with graph set $R_2^2(22)$ (Fig. 2). Mole-

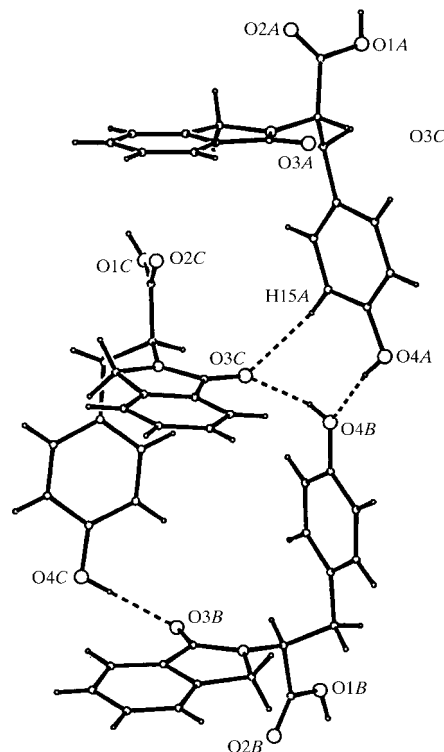


Figure 2
A view of the intermolecular interactions in (I), showing the interaction-intense $R_3^3(8)$ motif involving molecules *A*, *B* and *C*.

cule *A* interacts with this *B/C* dimer through an O4A—H4A···O4B—H4C···O3C relay [O4A···O4B 2.753 (5) Å], in combination with a C15A—H15A···O3C interaction [C15A···O3C 3.425 (6) Å], forming a hydrogen-bonded ring with graph set $R_3^3(8)$. The phenolic O4A—H4A group acts as a weak acceptor [C12B—H12B···O4Aⁱⁱ, with C12B···O4Aⁱⁱ 3.473 (5) Å; symmetry code: (ii) $x - \frac{1}{2}, \frac{1}{2} - y, 1 - z$] and as a donor in the O4A—H4A···O4B—H4C···O3C relay of interactions. Significant bending of the *para*-disubstituted atoms from the phenylene ring plane occurs in *A*, with C21A displaced from and *cisoid* to the aromatic plane by 0.163 (7) Å and O4A by 0.036 (7) Å. The C21A—C11A···C14A and C11A···C14A—O4A angles are bent by 174.1 (4) and 176.4 (3)°, respectively. This distortion about the C₆H₄ ring plane arises from the consequences of hydrogen bonding in the interaction-intense $R_3^3(8)$ motif as *A* maximizes its association with the *B/C* dimer.

The carbonyl O3A atom is an acceptor for two carboxylic acid O1A—H1A and O1B—H1B hydrogen-bond donors, O3B is an acceptor of a carboxylic acid O1C—H1C and a phenolic O4C—H4C donor, while O3C interacts through one short phenolic O4B—H4B···O3C hydrogen bond [O4B···O3C 2.607 (3) Å] and in combination with the C15A—H15A···O3C interaction. The carboxylate O2*n* atoms participate in weak intermolecular hydrogen bonding, the most important contact being C15C—H15C···O2B^v [C···O 3.569 (7) Å; symmetry code: (v) $-x, y - \frac{1}{2}, \frac{1}{2} - z$]. This weak participation of the carboxylate O2*n* atoms in hydrogen bonding also occurs in the L-threonine derivative and contrasts with the DL-threonine form, where centrosymmetric carboxylic O—H···O=C interactions assemble with graph set $R_2^2(8)$ (Gallagher *et al.*, 2000). The C—H···O interactions and C—H··· π_{arene} contacts complete the three-dimensional hydrogen bonding.

Crystal engineering studies continue to rely on stronger hydrogen bonds for the design and synthesis of three-dimensional structures (Aakeröy *et al.*, 1999). However, a fundamental understanding of the control and exploitation of *X*—H···*Y* and *X*—*Y*··· π_{arene} interactions (*X* = C, N or O; *Y* = N or O) remains an elusive goal. The conformational polymorphism of 5-methyl-2-[(2-nitrophenyl)amino]-3-thiophene-carbonitrile, a hexamorphic crystal system, with emphasis on the relative thermodynamic stability of the six polymorphs, has been described recently by Yu *et al.* (2000), serving to underline the difficulty in predicting crystal structures even in small molecules.

Experimental

The title compound was prepared by the overnight reaction of L-tyrosine and *o*-phthalaldehyde in refluxing acetonitrile (Allin *et al.*, 1996). Filtration of the hot solution and subsequent slow cooling of the filtrate allowed the isolation of large colourless crystals of (I) [m.p. 503–505 K (uncorrected)]. IR (KBr, ν_{OH} , cm⁻¹): 3339; ($\nu_{\text{C=O}}$, cm⁻¹): 1760, 1745, 1689; ¹H NMR (400 MHz, δ , *d*₆-DMSO, p.p.m.): 3.06 (*m*, 2H, CH₂), 4.43 (*br, s*, 2H, CH₂), 5.02 (*m*, 1H, CH), 6.59–7.02 (*m*, 4H, *para*-C₆H₄), 7.35–7.39, 7.44–7.48, 7.49–7.55, 7.56–7.58 (*m*, 4H, C₆H₄), 9.20 (*s*, 1H, OH), 10.48 (*s*, 1H, OH).

Crystal data

C₁₇H₁₅NO₄
M_r = 297.30
 Orthorhombic, *P*2₁2₁2₁
a = 10.7589 (9) Å
b = 17.8696 (17) Å
c = 22.6670 (18) Å
V = 4357.9 (7) Å³
Z = 12
D_x = 1.359 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 9.6–18.6°
 μ = 0.098 mm⁻¹
T = 294 (1) K
 Block, colourless
 0.36 × 0.36 × 0.28 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 8116 measured reflections
 4272 independent reflections
 2782 reflections with *I* > 2σ(*I*)
R_{int} = 0.009

θ_{max} = 25°
 h = -12 → 12
 k = 0 → 21
 l = 0 → 26
 3 standard reflections
 frequency: 120 min
 intensity decay: <1%

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.045
wR (*F*²) = 0.116
S = 1.02
 4272 reflections
 602 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.063P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.19 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.18 \text{ e } \text{Å}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick, 1997)
 Extinction coefficient: 0.0013 (3)

All atoms bound to C atoms were treated as riding, with *SHELXL97* (Sheldrick, 1997) defaults for C—H distances and with *U*_{iso}(H) = 1.5*U*_{eq}(C) for methyl H atoms and 1.2*U*_{eq}(C) for the remainder. The H atoms bound to O atoms were located from a difference Fourier map and subsequently treated as rigid rotating groups with *U*_{iso}(H) = 1.5*U*_{eq}(O). Examination of the structure of (I) with *PLATON* (Spek, 1998) showed that there were no solvent-accessible voids in the crystal lattice. A stereoview of the three molecules in the asymmetric unit of the unit cell has been deposited

Table 1

Selected geometric parameters (Å, °).

O1A—C1A	1.321 (5)	O3B—C3B	1.250 (5)
O2A—C1A	1.187 (5)	O4B—C14B	1.382 (5)
O3A—C3A	1.245 (5)	O1C—C1C	1.303 (5)
O4A—C14A	1.372 (5)	O2C—C1C	1.174 (5)
O1B—C1B	1.316 (5)	O3C—C3C	1.239 (5)
O2B—C1B	1.187 (6)	O4C—C14C	1.370 (6)
O1A—C1A—O2A	124.2 (5)	O3B—C3B—C4B	129.3 (4)
O1A—C1A—C2A	111.1 (4)	O4B—C14B—C13B	121.7 (5)
O2A—C1A—C2A	124.6 (4)	O4B—C14B—C15B	118.1 (4)
O3A—C3A—N1A	123.7 (4)	C13B—C14B—C15B	120.2 (5)
O3A—C3A—C4A	128.3 (4)	O1C—C1C—O2C	122.6 (5)
O4A—C14A—C13A	116.5 (4)	O1C—C1C—C2C	119.9 (4)
O4A—C14A—C15A	123.9 (4)	O2C—C1C—C2C	125.6 (4)
C13A—C14A—C15A	119.6 (5)	O3C—C3C—N1C	123.6 (4)
O1B—C1B—O2B	123.9 (5)	O3C—C3C—C4C	129.8 (4)
O1B—C1B—C2B	110.9 (5)	O4C—C14C—C13C	122.4 (5)
O2B—C1B—C2B	125.1 (5)	O4C—C14C—C15C	118.6 (5)
O3B—C3B—N1B	123.2 (4)	C13C—C14C—C15C	119.0 (5)
O1A—C1A—C2A—C21A	-49.8 (5)	N1B—C2B—C21B—C11B	-63.6 (6)
O2A—C1A—C2A—C21A	132.3 (5)	C1B—C2B—C21B—C11B	172.4 (4)
N1A—C2A—C21A—C11A	-71.7 (5)	O1C—C1C—C2C—C21C	-53.4 (6)
C1A—C2A—C21A—C11A	162.8 (4)	O2C—C1C—C2C—C21C	128.3 (6)
O1B—C1B—C2B—C21B	-65.5 (5)	N1C—C2C—C21C—C11C	-52.5 (5)
O2B—C1B—C2B—C21B	113.0 (6)	C1C—C2C—C21C—C11C	-177.4 (4)

Table 2

Hydrogen-bonding geometry (Å, °).

Cg1 is the centroid of the C11C–C16C ring.

<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>
O1A–H1A···O3A ⁱ	0.82	1.84	2.652 (4)	169
O1B–H1B···O3A ⁱⁱ	0.82	2.00	2.716 (5)	147
O1C–H1C···O3B ⁱⁱⁱ	0.82	1.89	2.675 (5)	159
O4A–H4A···O4B	0.82	1.94	2.753 (5)	170
O4B–H4B···O3C	0.82	1.85	2.607 (4)	152
O4C–H4C···O3B	0.82	2.00	2.809 (4)	171
C10C–H10E···O1A ^{iv}	0.97	2.50	3.284 (5)	138
C15A–H15A···O3C	0.93	2.54	3.425 (6)	160
C12B–H12B···O4A ⁱⁱ	0.93	2.66	3.473 (5)	147
C15C–H15C···O2B ^v	0.93	2.65	3.569 (7)	173
C5A–H5A···Cg1 ^{vi}	0.93	2.86	3.716 (5)	153

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

as a supplementary diagram. Refinement of the full data set with all Friedel reflections not unexpectedly gave an inconsistent Flack (1983) parameter [0.6 (16)] for this light-atom structure; for the final refinement, the Friedel reflections were merged.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992); cell refinement: *CAD-4-PC Software*; data reduction: *DATRD2* in *NRCVAX96* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *NRCVAX96* and *SHELXL97*; molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEX* (McArdle, 1995) and *PLATON*; software used to prepare material for publication: *NRCVAX96*, *SHELXL97* and *PREP8* (Ferguson, 1998).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1414). An additional figure is also available. Services for accessing these data are described at the back of the journal.

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