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Key indicators

Single-crystal X-ray study
 T = 294 K
 Mean σ (C–C) = 0.005 Å
 Disorder in main residue
 R factor = 0.044
 wR factor = 0.080
 Data-to-parameter ratio = 7.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

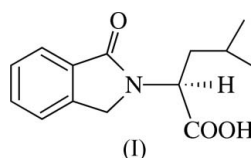
(2S)-4-Methyl-2-(1-oxo-1H-2,3-dihydro-isoindol-2-yl)pentanoic acid

The title compound, C₁₄H₁₇NO₃, exhibits carboxylic acid group disorder about the C–CO₂ axis, with site occupancies of 0.79 (5):0.21 (5). Molecules are linked by intermolecular O–H···O=C_{iso}, C–H···O=C_{iso} and C–H··· π (arene) interactions (iso = isoindolinone).

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Comment

The majority of structurally determined phthalimidine systems are either *N*-substituted or have a hydroxy substituent at the 3-position (McNab *et al.*, 1997; Mukherjee *et al.*, 2000). The title compound, (I), synthesized from L-leucine and *ortho*-phthalaldehyde (Allin *et al.*, 1996), forms part of a structural study of phthalimidines (Brady *et al.*, 1998; Gallagher *et al.*, 2000; Gallagher & Brady, 2000; Gallagher & Murphy, 2001).



The molecular structure of (I) is depicted in Fig. 1 (*S* configuration) and selected dimensions are given in Table 1. The geometric data are normal (McNab *et al.*, 1997) and in agreement with expected values (Allen, 2002). The five- and six-membered rings of the isoindole group are coplanar [dihedral angle between rings = 1.0 (2)°], and the isoindolinone atom O3 is 0.022 (5) Å from the C₄N ring plane; this ring is oriented at 82.5 (5)° to the major orientation of the CCO₂ plane (O1A/O2A/C1/C2).

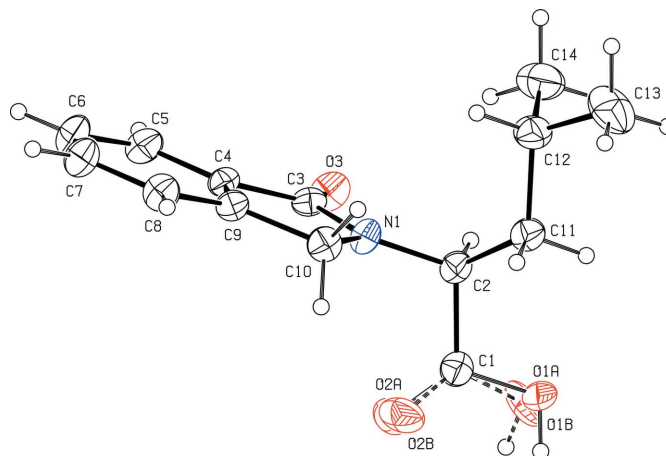


Figure 1
 A view of (I), with the atomic numbering scheme; displacement ellipsoids are drawn at the 30% probability level. Both disorder components are shown.

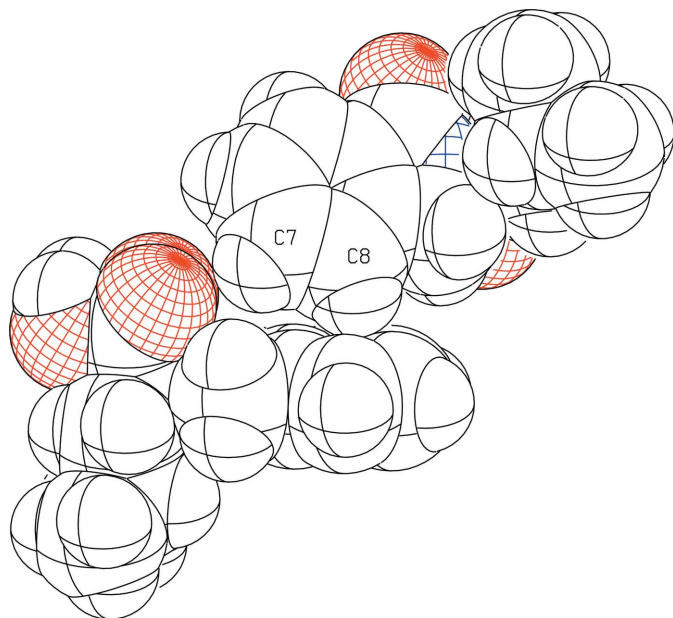


Figure 2
Two molecules of (I), with atoms depicted as their van der Waals spheres, with C7 (C—H···Oⁱⁱⁱ contact) and C8 [C—H··· π (arene)ⁱⁱⁱ] labels.

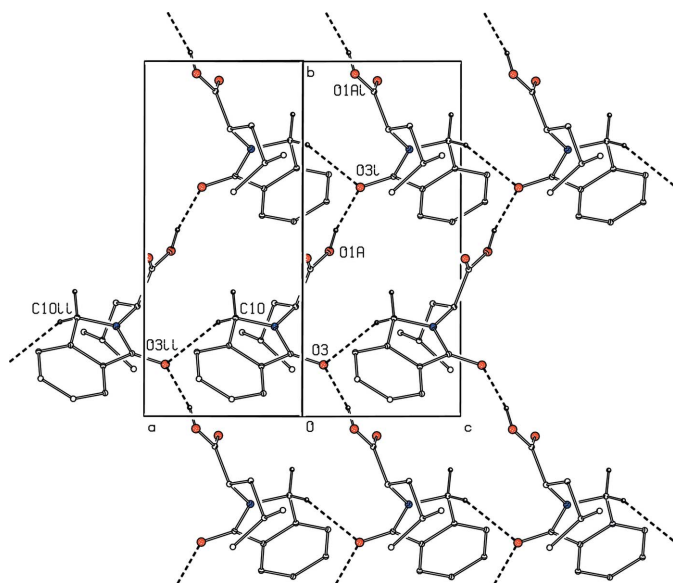


Figure 3
A view of the $C(7)C(5)[R_4^3(20)]$ sheet in (I) with the unit-cell outline (symmetry codes as in Table 2); H atoms not involved in hydrogen bonding have been omitted for clarity.

Molecules of (I) exhibit CO₂H group disorder about the C—CO₂ axis with site occupancies of 0.79 (5):0.21 (5) for the major/minor sites, respectively. Conventional CO₂H dimeric hydrogen bonding [$R_2^2(8)$ ring] is not present as a requirement of symmetry; rather, the primary hydrogen bonding as an (\cdots O—H···O—H···)_n chain along [010] involving O1A/B—H1A/B···O3ⁱ (Table 1) is described by a $C(7)$ motif (Grell *et al.*, 1999). The closest H atoms to the carbonyl O2A/B are at contact distances, *e.g.* H7···O2Aⁱⁱⁱ is 2.71 Å, with C7—H7···O2Aⁱⁱⁱ = 136° (symmetry codes iii as in Table 2). Disorder is facilitated on geometric grounds as O2 can rotate

about the C1—C2 axis without greatly affecting the O1A/B—H1A/B···O3ⁱ interaction distance (Fig. 2).

Combination of the O—H···O=C_{iso} C(7) motif with a $C(5)$ motif (from C10—H10A···O3ⁱⁱ) generates a two-dimensional sheet comprising $R_4^3(20)$ rings as $C(7)C(5)[R_4^3(20)]$; modest (arene)C—H··· π (arene) interactions (Nishio, 2004) link these sheets (Fig. 3 and Table 2).

Compound (I) and the L-norvaline derivative, (II), C₁₃H₁₅NO₃, (2*S*)-2-(1-oxo-1*H*-2,3-dihydroisindol-2-yl)-pentanoic acid (Gallagher & Brady, 2000), both crystallize in space group $P2_12_12_1$ with similar cell dimensions. The corresponding atom coordinates and molecular conformations are comparable and hence the crystal structures are isomorphous. Molecules of (I) and (II) differ in their respective alkyl chains with the (CH₃)₂CHCH₂— group in (I) occupying a similar volume as the disordered CH₃CH₂CH₂— group in (II). The solid-state (KBr disk) C=O stretching vibrations are similar, 1736, 1638 cm⁻¹ in (I) and 1730, 1649 cm⁻¹ in (II), highlighting the analogous environments of both C=O groups in (I) and (II).

Experimental

The title compound (I) was prepared by the overnight reaction of L-leucine and *o*-phthalaldehyde in refluxing CH₃CN under N₂ (Allin *et al.*, 1996). Filtration of the hot solution and subsequent slow cooling of the filtrate allowed the isolation of block-like colourless crystals. M.p. 485–487 K (uncorrected).

Crystal data

C₁₄H₁₇NO₃
M_r = 247.29
Orthorhombic, $P2_12_12_1$
a = 5.8790 (5) Å
b = 12.5223 (16) Å
c = 18.029 (3) Å
V = 1327.3 (3) Å³

Z = 4
D_x = 1.237 Mg m⁻³
Mo K α radiation
 μ = 0.09 mm⁻¹
T = 294 (1) K
Block, colourless
0.35 × 0.25 × 0.15 mm

Data collection

Enraf–Nonius CAD-4
diffractometer
 ω -2 θ scans
Absorption correction: none
2394 measured reflections
1373 independent reflections

889 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
 θ_{\max} = 25.0°
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
R[F² > 2 σ (F²)] = 0.044
wR(F²) = 0.080
S = 1.00
1373 reflections
185 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0312P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
(Δ/σ)_{max} < 0.001
 $\Delta\rho_{\max}$ = 0.13 e Å⁻³
 $\Delta\rho_{\min}$ = -0.13 e Å⁻³
Extinction correction: SHELXL97
Extinction coefficient: 0.013 (2)

Table 1

Selected torsion angles (°).

C3—N1—C2—C11	133.9 (3)	O3—C3—N1—C2	-1.6 (5)
C3—N1—C2—C1	-97.4 (4)	O1A—C1—C2—C11	-51.2 (9)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1A-H1A\cdots O3^i$	0.82	1.83	2.634 (9)	168
$C10-H10A\cdots O3^{ii}$	0.97	2.54	3.366 (4)	144
$C8-H8\cdots Cg1^{iii}$	0.93	2.74	3.473 (4)	137
$C2-H2\cdots O3$	0.98	2.38	2.812 (4)	106

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

In the absence of significant anomalous dispersion effects, Friedel equivalents were merged prior to the final refinement cycles. The absolute configuration can be inferred from the known absolute configuration of the L-leucine starting material. H atoms were treated as riding atoms using the *SHELXL97* (Sheldrick, 1997) defaults [at 294 (1) K], with C–H distances from 0.93 to 0.98 Å and O–H = 0.82 Å, and with $U_{iso}(H)$ from 1.2 to 1.5 times $U_{eq}(C,O)$.

Data collection: *CAD-4* (Enraf–Nonius, 1992); cell refinement: *SET4* and *CELDIM* (Enraf–Nonius, 1992); 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* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *WORDPERFECT* macro *PREP8* (Ferguson, 1998).

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