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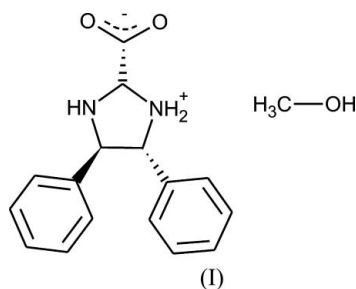
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Key indicators

Single-crystal X-ray study
 $T = 130$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.046
 wR factor = 0.091
Data-to-parameter ratio = 7.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(4*R*,5*R*)-4,5-Diphenylimidazolidinium-2-
carboxylate methanol solvate**The title compound, $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_2 \cdot \text{CH}_4\text{O}$, exists in the solid
state in a zwitterionic form. Intermolecular hydrogen bonds
assemble the molecules into a two-dimensional network.Received 28 June 2006
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Comment

Imidazolidine-2-carboxylic acid is an α,α' -diamino acid, which can be considered as an analogue of proline in which the α -methylene group in the pyrrolidine ring has been substituted by an NH group. Very little is known about the structure of this type of compound. Our search of the Cambridge Structural Database (Version 5.27, plus January 1 2006 update; Allen, 2002) revealed only one crystal structure of an aliphatic α,α' -diamino acid, a derivative of 1,2-dihydroquinazoline (Hoefnagel *et al.*, 1993). It has been shown recently that optically active imidazolidine-2-carboxylic acids can be employed as organocatalysts in asymmetric synthesis (Halland *et al.*, 2002, 2003). These compounds are easily formed in a well known condensation reaction of α -diamines with aldehydes (Hofmann, 1953; Fieser & Fieser, 1967) in which glyoxalic acid is used as an aldehydic substrate. To obtain more information about the structure and assembly mode of imidazolidine-2-carboxylic acids, we prepared (4*R*,5*R*)-4,5-diphenylimidazolidine-2-carboxylic acid and determined the crystal structure of its methanol solvate, (I).



The asymmetric unit of (I) is shown in Fig. 1. The acid crystallizes in a zwitterionic form with a proton transferred from the carboxylic group to the imidazolidine N1 atom. The five-membered ring adopts a slightly distorted envelope conformation with C4 as the flap and this allows for the vicinal *trans*-oriented phenyl substituents to be equatorially positioned (Table 1). As a result of a difference in the protonation state of the imidazolidine N atoms, an additional chiral center, at the C2 atom, exists in the zwitterionic form of (I). This new center has the *S* configuration in the crystal structure whereas in solution, due to an easy proton transfer, the *RRS* and *RRR* diastereomers have to be in equilibrium. The carboxylate group orientation in the molecule is determined by the

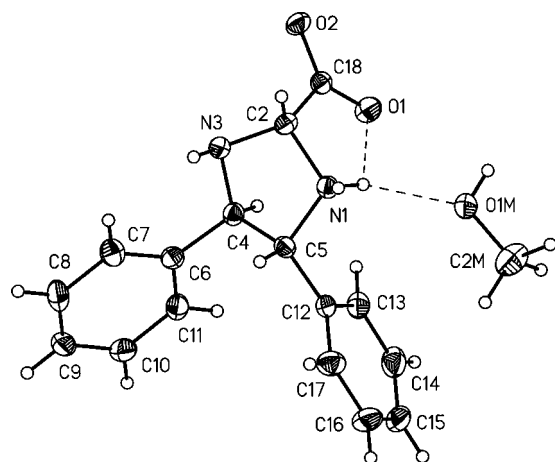


Figure 1
The asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The dashed lines indicate hydrogen bonds.

intramolecular N1—H1N_A···O1 hydrogen bond (Table 2), which is a part of a bifurcated hydrogen bond.

The crystal packing in (I) is shown in Fig. 2*a*. All O—H and N—H donors are involved in hydrogen bonding. The imidazolidinium-2-carboxylate molecules are connected *via* N1—H1N_B···O2(1 - *x*, *y* - ½, ½ - *z*) and N3—H3N···O1(*x* - 1, *y*, *z*) hydrogen bonds into a two-dimensional assembly parallel to the (001) plane (Table 2 and Fig. 2*b*). The channels within this assembly, which run parallel to the *a* axis, are occupied by the methanol molecules which act as both hydrogen-bond donors and acceptors (Table 2 and Fig. 2*b*).

Experimental

Compound (I) was prepared analogously to the procedure reported by Halland *et al.* (2003). To a solution of (*R,R*)-1,2-diphenylethane-1,2-diamine (0.119 g, 0.56 mmol) in 5 ml CH₂Cl₂ at 273 K was added glyoxalic acid monohydrate (0.052 g, 0.56 mmol) dissolved in 2.5 ml MeOH and the reaction mixture was stirred for 15 h at room temperature. The reaction mixture was transferred to a small beaker and the solvent allowed to evaporate slowly. Colourless crystals, which precipitated, were suitable for X-ray analysis. The crystals were unstable and, when left in air, decomposed within a few days with loss of solvent.

Crystal data

C ₁₆ H ₁₆ N ₂ O ₂ ·CH ₄ O	<i>Z</i> = 4
<i>M_r</i> = 300.35	<i>D_x</i> = 1.289 Mg m ⁻³
Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁	Mo <i>K</i> α radiation
<i>a</i> = 5.9624 (4) Å	<i>μ</i> = 0.09 mm ⁻¹
<i>b</i> = 8.8288 (6) Å	<i>T</i> = 130 (2) K
<i>c</i> = 29.4086 (19) Å	Prism, colorless
<i>V</i> = 1548.09 (18) Å ³	0.4 × 0.3 × 0.05 mm

Data collection

Kuma KM-4-CCD <i>κ</i> -geometry diffractometer	1616 independent reflections
<i>ω</i> scans	1528 reflections with <i>I</i> > 2σ(<i>I</i>)
Absorption correction: none	<i>R</i> _{int} = 0.041
11990 measured reflections	<i>θ</i> _{max} = 25.0°

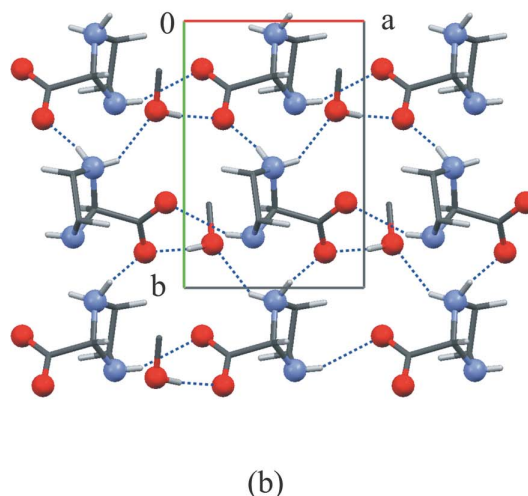
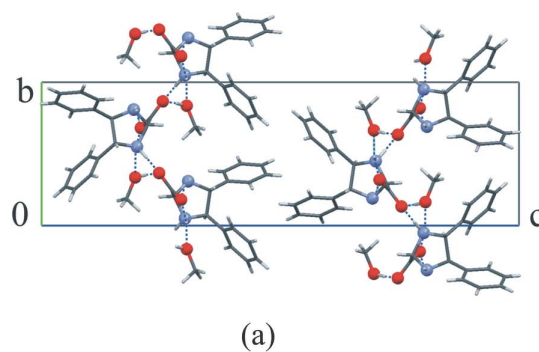


Figure 2
The crystal structure of (I), showing (a) the crystal packing viewed down the *a* axis and (b) the two-dimensional network of hydrogen-bonded (dashed lines) molecules (the phenyl rings and the methyl H atoms have been omitted for clarity).

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.046
wR(*F*²) = 0.091
S = 1.16
 1616 reflections
 218 parameters
 H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0376P)^2 + 0.5565P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 0.26 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.18 \text{ e \AA}^{-3}$

Table 1
Selected geometric parameters (Å, °).

O1—C18	1.241 (3)	C2—N3	1.455 (4)
O2—C18	1.265 (3)	C2—C18	1.541 (4)
N1—C5	1.502 (4)	N3—C4	1.475 (4)
N1—C2	1.528 (3)		
C5—N1—C2—N3	5.4 (3)	N3—C4—C5—N1	-38.5 (3)
N1—C2—N3—C4	-30.2 (3)	N1—C2—C18—O1	12.7 (3)
C2—N3—C4—C5	42.9 (3)	N3—C2—C18—O2	74.5 (3)
C2—N1—C5—C4	20.0 (3)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1NA\cdots O1$	0.84 (3)	2.17 (3)	2.687 (3)	120 (3)
$N1-H1NA\cdots O1M$	0.84 (3)	2.11 (3)	2.816 (3)	142 (3)
$N1-H1NB\cdots O2^i$	0.97 (3)	1.74 (4)	2.686 (3)	167 (3)
$N3-H3N\cdots O1^{ii}$	0.85 (3)	2.33 (3)	3.139 (3)	158 (3)
$O1M-H1M\cdots O2^{iii}$	0.98 (4)	1.71 (4)	2.683 (3)	171 (4)

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

In the absence of significant anomalous scattering effects, Friedel pairs were averaged. The chirality of atoms C4 and C5 were assigned from the known hand of the starting material. All H atoms of OH and NH groups were located in electron-density difference maps and were freely refined. H atoms bonded to C atoms were placed at calculated positions, with $C-H = 0.93-0.96$ Å, and were refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation*

Operation Manual (Siemens, 1989) and *MERCURY* (Version 1.4; Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL97*.

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