

4,5-Diphenyl-1*H*-imidazole-1-acetic acidShan Gao,^a Hui Zhao,^a Li-Hua
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Key indicators

Single-crystal X-ray study

T = 295 K

Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$

R factor = 0.075

wR factor = 0.244

Data-to-parameter ratio = 10.0

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

In the crystal structure of the title compound, $\text{C}_{17}\text{H}_{14}\text{N}_2\text{O}_2$, the 4-phenyl ring is twisted by $24.4(3)^\circ$ and the 5-phenyl ring by $72.6(2)^\circ$ with respect to the imidazole ring. Four adjacent molecules are linked by short $\text{O}-\text{H}\cdots\text{N}$ [$\text{O}\cdots\text{N} = 2.602(6) \text{ \AA}$] interactions into a hydrogen-bonded tetrameric entity. The tetramers are stacked over each other along the short *c* axis [$5.663(3) \text{ \AA}$].

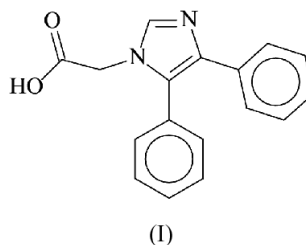
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Comment

Few imidazole-substituted acetic acids have been crystallographically authenticated. Imidazole-4-acetic acid, a chemical of biochemical importance, exists as a hydrated zwitterion (Okabe & Hiyashi, 1999); the compound forms salts with hydrochloric acid (Jones & Pauling, 1976) and picric acid (In *et al.*, 1997). The 1-imidazole analogue also exists as a zwitterion (Lopez *et al.*, 1996). The title diphenyl-substituted imidazole-1-acetic acid, (I) (Fig. 1), exists as a neutral molecule; four adjacent molecules are linked by hydrogen bonds into a hydrogen-bonded tetrameric entity. The tetramers are stacked over each other along the *c* axis. The twists of the two phenyl rings are similar to the twists found in 4,5-diphenylimidazole itself (Stibrany *et al.*, 2004).



The energy of the geometry-optimized neutral structure was calculated to be $197 \text{ kcal mol}^{-1}$ ($1 \text{ kcal mol}^{-1} = 4.184 \text{ kJ mol}^{-1}$) more stable than the crystal structure. The twists of the two phenyl rings with respect to the imidazole ring between the calculated and X-ray structures are nearly identical, and the only noticeable difference in geometry between them is centered on the carboxyl fragment. The difference appears to be a necessary condition for four adjacent molecules to assemble into a hydrogen-bonded supramolecular entity in the crystal structure (Fig. 2).

Experimental

Equimolar quantities of glycine, dibenzoyl and sodium hydroxide were dissolved in ethanol. The mixture was refluxed for 4 h. The filtered solution was acidified with concentrated hydrochloric acid. Colorless crystals separated from the solution after several days.

Crystal data

C₁₇H₁₄N₂O₂
M_r = 278.30
 Tetragonal, *I*4̄
a = 23.048 (7) Å
c = 5.663 (3) Å
V = 3008 (2) Å³
Z = 8
D_x = 1.229 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 13 806 reflections
 $\theta = 3.5\text{--}27.5^\circ$
 $\mu = 0.08\text{ mm}^{-1}$
T = 295 (2) K
 Block, colorless
 0.42 × 0.31 × 0.26 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
 ω scans
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
T_{min} = 0.800, *T_{max}* = 0.979
 14 058 measured reflections

1903 independent reflections
 1659 reflections with *I* > 2σ(*I*)
R_{int} = 0.033
 $\theta_{\text{max}} = 27.5^\circ$
h = -29 → 29
k = -29 → 29
l = -7 → 6

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.075
wR (*F*²) = 0.244
S = 1.25
 1903 reflections
 191 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1675P)^2 + 0.0654P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.72\text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.36\text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1—C1	1.285 (6)	C6—C7	1.397 (6)
O2—C1	1.192 (8)	C7—C8	1.384 (6)
N1—C3	1.337 (6)	C8—C9	1.384 (7)
N1—C5	1.384 (6)	C9—C10	1.364 (8)
N1—C2	1.466 (6)	C10—C11	1.399 (6)
N2—C3	1.311 (7)	C12—C13	1.398 (5)
N2—C4	1.394 (5)	C12—C17	1.403 (6)
C1—C2	1.538 (8)	C13—C14	1.390 (7)
C4—C5	1.371 (6)	C14—C15	1.375 (7)
C4—C12	1.473 (6)	C15—C16	1.384 (7)
C5—C6	1.486 (5)	C16—C17	1.379 (7)
C6—C11	1.387 (6)		
C2—N1—C3	125.2 (4)	C7—C6—C11	119.4 (4)
C2—N1—C5	125.2 (3)	C5—C6—C7	120.6 (4)
C3—N1—C5	107.6 (4)	C6—C7—C8	119.8 (4)
C3—N2—C4	106.5 (4)	C7—C8—C9	120.3 (5)
O1—C1—O2	126.2 (6)	C8—C9—C10	120.4 (4)
O1—C1—C2	111.4 (5)	C9—C10—C11	120.1 (4)
O2—C1—C2	122.4 (5)	C6—C11—C10	119.9 (4)
N1—C2—C1	109.5 (5)	C13—C12—C17	117.7 (4)
N1—C3—N2	111.6 (4)	C4—C12—C13	121.3 (4)
N2—C4—C5	108.2 (4)	C4—C12—C17	121.0 (4)
N2—C4—C12	122.2 (4)	C12—C13—C14	120.7 (4)
C5—C4—C12	129.7 (3)	C13—C14—C15	120.3 (4)
N1—C5—C4	106.1 (3)	C14—C15—C16	120.0 (5)
N1—C5—C6	121.8 (4)	C15—C16—C17	120.0 (5)
C4—C5—C6	132.1 (4)	C12—C17—C16	121.2 (4)
C5—C6—C11	120.0 (4)		

For the geometry-optimization calculations with *HyperChem* (Hypercube, 2000), the starting structure was taken from the crystal structure, and this was optimized at the *PM3* level. Whether the acid H atom was on the carboxyl group or the imidazole N atom could not be decided from the diffraction measurement. The first set of geometry-optimization calculations was performed on the zwitterionic structure, followed by calculations on the neutral structure. The neutral structure was found to be more stable by about 27 kcal mol⁻¹ (*kT* is approximately 0.6 kcal mol⁻¹ at room temperature). The H atoms were placed in calculated positions [aromatic C—H = 0.93 Å and aliphatic C—H = 0.97 Å; *U_{iso}*(H) = 1.2*U_{eq}*(C)], and were included

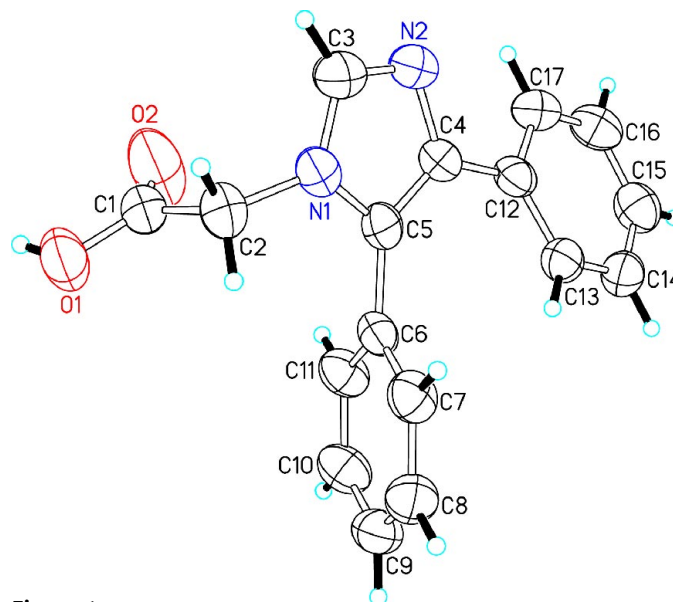


Figure 1
 ORTEPII (Johnson, 1976) plot of (I), with ellipsoids drawn at the 50% probability level. [Symmetry code: (i) $\frac{1}{2} - x, 1 - y, \frac{1}{2} + z$.]

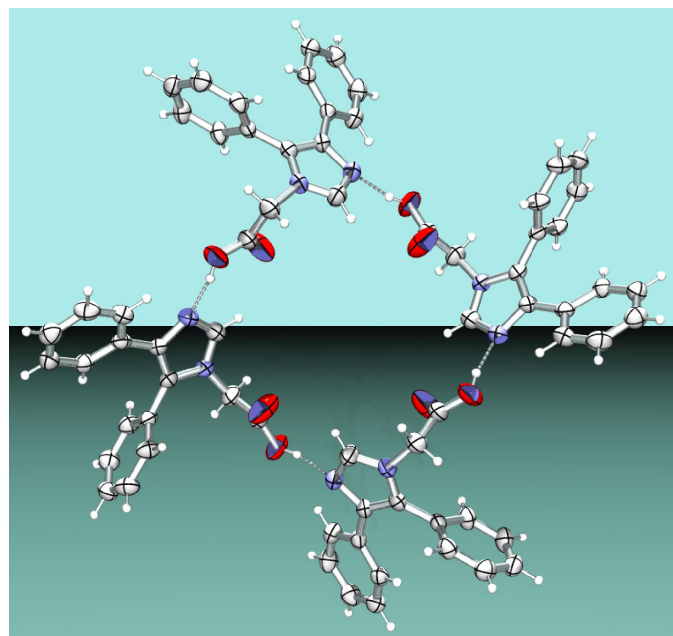


Figure 2
 POV-Ray for Windows (Cason, 2002)/ORTEPII (Johnson, 1976) plot of the hydrogen-bonded tetramer of (I) [O...N = 2.602 (6) Å] viewed along the *c* axis. Hydrogen bonds are shown as dashed lines.

in the refinement in the riding-model approximation. The acid H atom could not be located from a difference Fourier map and was generated geometrically by allowing the OH unit to rotate so as to fit the electron density. As there are no heavy atoms that will permit the refinement of the Flack (1983) parameter, Friedel pairs were merged.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *POV-Ray for Windows* (Cason, 2002); software used to prepare material for publication: *SHELXL97*.

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