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A second polymorph of 4,5-diphenyl-1*H*-imidazole

Robert T. Stibrany, Harvey J. Schugar and Joseph A. Potenza*

Department of Chemistry and Chemical Biology, Rutgers, The State University of New Jersey, 610 Taylor Road, Piscataway, New Jersey 08854, USA

Correspondence e-mail: potenza@rutchem.rutgers.edu

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.063
wR factor = 0.185
Data-to-parameter ratio = 10.5

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

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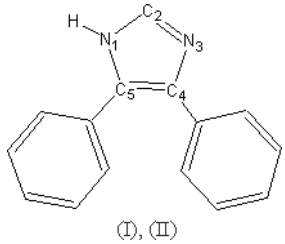
The title compound, $C_{15}H_{12}N_2$, $(II)_{(499)}$, contains two molecules in the asymmetric unit and is a polymorphic form of a previously reported structure, $(I)_{(495)}$, of the same molecule. The molecules in both polymorphs exhibit nearly identical bond distances, but show significantly different phenyl/imidazole dihedral angles and pack differently in the crystalline state. In each polymorph, strong hydrogen-bonded polymer chains, involving the N atoms of the imidazole fragments in adjacent molecules, are formed.

Comment

Substituted 4,5-diphenylimidazoles are known to be active pharmacologically. For example, a mevalonate derivative containing a 4,5-diphenyl-1*H*-imidazole group has been shown to reduce cholesterol levels in rats (Harris *et al.*, 1992). From the standpoints of drug performance and of intellectual property, polymorphism is an important aspect of pharmacologically active compounds. As an example, sertraline hydrochloride, the active drug in Zoloft™, has been found to have 17 different polymorphic forms (Almarsson *et al.*, 2003). One of our current interests is to use monodentate imidazoles and benzimidazoles as building blocks to synthesize bis-chelating ligands which are useful for preparing metal-ion complexes with unusual coordination geometries and properties (Stibrany *et al.*, 2004). Several methods have been reported for joining imidazole fragments to prepare such ligands (Gorun *et al.*, 1996; Sugimoto *et al.*, 1998). In this study, we report a second polymorph of the monodentate building block 4,5-diphenylimidazole.



(I), (II)

The structure of $(II)_{(499)}$ contains two 4,5-diphenyl-1*H*-imidazole molecules in the asymmetric unit (Fig. 1), in contrast to the polymorph reported previously, $(I)_{(495)}$, which contains only one such molecule [refcode OCUSUA (Stibrany *et al.*, 2001) in the Cambridge Structural Database (Version 5.24; Allen, 2002)]. Here, we use the nomenclature encouraged by Bernstein (2002), in which polymorphs are designated in order of discovery by increasing Roman numerals followed by their melting point in Kelvin. Bond lengths in the imidazole fragments of $(I)_{(495)}$ and $(II)_{(499)}$ agree well (Table 2). In addition,

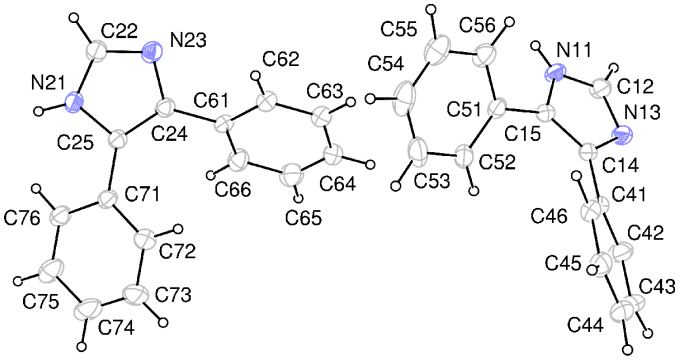


Figure 1
The contents of the asymmetric unit of $(II)_{(499)}$, showing 25% probability displacement ellipsoids and the atom-numbering scheme.

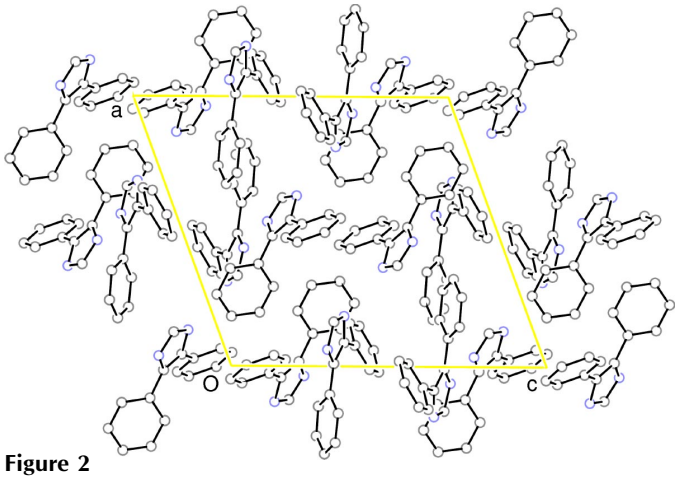


Figure 2
View, along the *b* axis, of the contents of the unit cell of $(II)_{(499)}$. H atoms have been omitted for clarity.

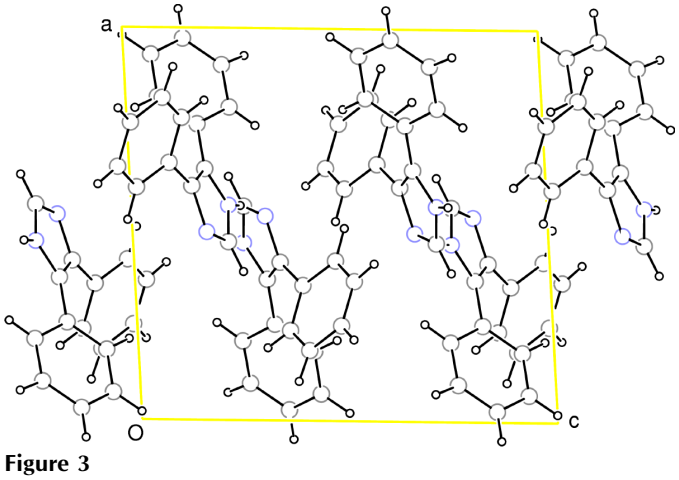


Figure 3
View, along the *b* axis, of the contents of the unit cell of $(I)_{(495)}$.

the imidazole (im) and phenyl (ph) fragments in both polymorphs are planar to within 0.01 Å, as expected. From a molecular perspective, the polymorphs differ primarily in the relative orientation of the ph and im fragments. In $(II)_{(499)}$, the ph/im dihedral angles are 35.81 (8) and 42.71 (8)° for one molecule, and 34.22 (11) and 41.15 (12)° for the second unique molecule. A greater difference in these angles is observed for polymorph $(I)_{(495)}$, in which the phenyl rings are tilted by 29.79 (6) and 42.75 (8)° with respect to the imidazole plane.

In crystal structure of $(II)_{(499)}$ (Fig. 2), one of the unique molecules (*A*) is related to the others by centers of symmetry at $(0, \frac{1}{2}, 0)$ and by the *n*-glide plane symmetry operations. The second unique molecule (*B*) is related similarly by centers of symmetry at $(\frac{1}{2}, \frac{1}{2}, 0)$ and by the *n*-glide plane operations. When coupled with translations along the **b** direction, a structure results which may be viewed as consisting of slightly overlapping, alternating slabs of *A* and *B* molecules parallel to the $(\bar{1}01)$ planes. *A*- and *B*-type molecules are linked to each other along the [101] direction by strong N—H···N hydrogen bonds (Table 1) to form infinite —*A*—*B*— chains. Intermolecular C—C and C—N distances suggest that the molecules are otherwise linked primarily by C—H···π interactions, with little, if any, π stacking.

In crystals of polymorph $(I)_{(495)}$ (space group $P2_1/c$, one molecule per asymmetric unit; Fig. 3), molecules related by the screw axes form columns along the **b** direction which are related to each other by the centers of symmetry and by the *c*-glide-plane symmetry operations. Within a given column, adjacent molecules are linked by N—H···N hydrogen bonds to form infinite chains along *b*. As with polymorph $(II)_{(499)}$, intermolecular interactions exclusive of hydrogen bonds appear to be dominated by C—H···π interactions, with little evidence to suggest π stacking.

Experimental

4,5-Diphenyl-1*H*-imidazole was synthesized by the benzoin condensation method, as reported by Bredereck & Theillig (1953). In that report, a melting point of 504 K was obtained for the product, which was recrystallized from diethyl malonate. Polymorph $(I)_{(495)}$ was obtained by recrystallization from 2-propanol, while the current polymorph, $(II)_{(499)}$, was obtained by recrystallizing $(I)_{(495)}$ from acetonitrile (m.p. 499 K).

Crystal data

$C_{15}H_{12}N_2$
M_r = 220.27
Monoclinic, $P2_1/n$
a = 13.3716 (7) Å
b = 13.0984 (8) Å
c = 14.6909 (12) Å
β = 110.343 (4)°
V = 2412.6 (3) Å^3
Z = 8
D_x = 1.213 Mg m^{-3}
D_m = 1.20 (1) Mg m^{-3}
D_m measured by flotation in a mixture of carbon tetrachloride and cyclohexane
Mo $K\alpha$ radiation
Cell parameters from 960 reflections
θ = 3.0–23.6°
μ = 0.07 mm^{-1}
T = 296 (1) K
Prism, colorless
0.55 × 0.44 × 0.26 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Blessing, 1995)
T_{min} = 0.783, T_{max} = 0.980
15100 measured reflections
4234 independent reflections
3444 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 25.0°
$h = -15 \rightarrow 15$
$k = -15 \rightarrow 13$
$l = -17 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.185
S = 1.00
4234 reflections
403 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.1214P)^2 + 0.4106P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N21—H21N···N13^{i}	0.86 (3)	2.09 (3)	2.917 (3)	160 (3)
N11—H11N···N23^{ii}	0.83 (3)	2.05 (3)	2.884 (3)	178 (3)

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

Table 2
Bond lengths (Å) in the imidazole fragments of polymorphs (I) and (II) (molecules *A* and *B*).

	(I)	(II), molecule *A*	(II), molecule *B*
N1—C2	1.340 (2)	1.334 (3)	1.336 (3)
C2—N3	1.314 (2)	1.317 (3)	1.312 (3)
N3—C4	1.385 (2)	1.391 (3)	1.383 (3)
C4—C5	1.374 (2)	1.373 (3)	1.369 (3)
N1—C5	1.376 (2)	1.378 (3)	1.378 (3)

Data collection: *SMART-WNT*(2000) (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2000); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996) and *ORTEP*-32 (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 2000).

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