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2-Oxo-1,2-dihydropyridine-6-carboxylic Acid

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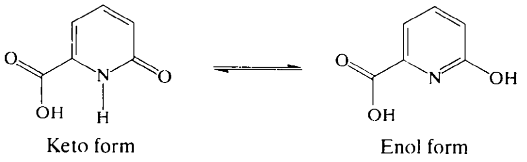
Abstract

The molecular structure of the title compound, $C_6H_5NO_3$, clearly indicates the keto–enol tautomerism of the CONH moiety, although the keto form is more preferable than the enol form. Two molecules are connected by two N—H···O hydrogen bonds around an inversion center to form a dimer, and the dimers are linked in a planar fashion by O—H···O hydrogen bonds to form a sheet.

Comment

2-Pyridone derivatives have been extensively studied since they cause base mispairing and enzymatic reactions related to bifunctional catalysis (Beak *et al.*, 1976). Theoretical analysis and NMR measurements have been performed for these compounds (Kuzuya *et al.*, 1984; Facelli *et al.*, 1992), and the crystal structures of some 2-pyridone derivatives have been determined previously (Low & Wilson, 1983; Kvick & Booles, 1972). In order to compare the molecular structure of the title compound, 2-oxo-1,2-dihydropyridine-6-carboxylic acid, (I), with the 2-pyridone derivatives, the data were collected at 200 K.



(I)

The molecular structure of (I) with atomic numbering is shown in Fig. 1 and the crystal structure viewed along the *b* axis is shown in Fig. 2. There are two considerably strong hydrogen bonds (N—H···O) between the two molecules around an inversion center, forming a dimer [N1···O7(1 − *x*, 2 − *y*, 1 − *z*) 2.873 (3) Å]. The H1 atom, confirmed from a difference Fourier map, is clearly bonded to the N atom of the six-membered ring. This indicates that the molecule is not an enol, but is instead in the keto form. However, the C2—O7 distance is longer than C8—O9 and shorter than C8—O10. Moreover, N1—C2 is shorter than a usual N—C bond (1.469 Å; *International Tables for Crystallography*, 1974, Vol. C). These distances suggest that the molecule adopts partly the enol form. The molecule is approximately planar, excluding the carboxyl group (r.m.s. deviation of fitted atoms is 0.008 Å). The C5—C6—C8—O9 torsion angle is −15.1 (5)°. These parameters are essentially the same as those of the related derivatives.

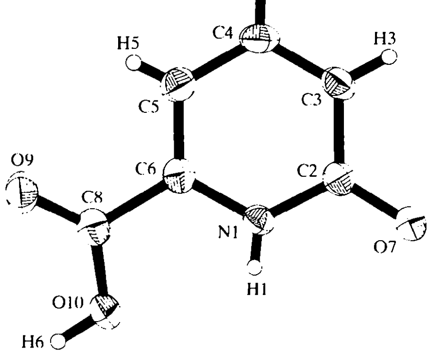


Fig. 1. The molecular structure of (I). Displacement ellipsoids are scaled to enclose 50% probability levels.

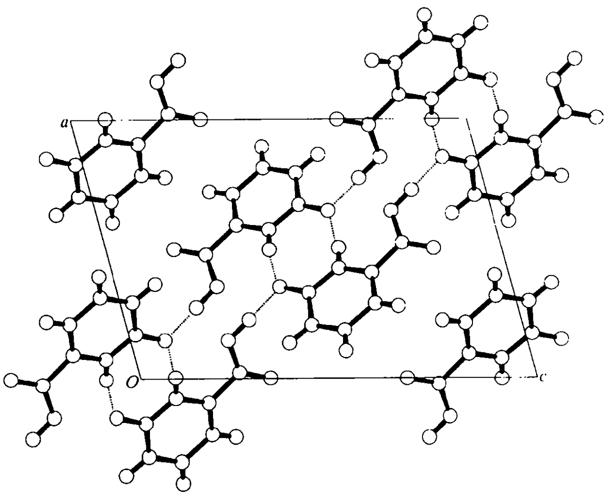


Fig. 2. The crystal structure of (I) along the *b* axis. Hydrogen bonds are shown as dotted lines.

The O10 atom of the carboxy group forms a strong hydrogen bond with O7 of the neighboring molecule at ($\frac{1}{2}+x$, $\frac{3}{2}-y$, $\frac{1}{2}+z$) [O10···O7 2.590 (3) Å]. There is a considerably short contact between O9 and H3 of another neighboring molecule at ($\frac{1}{2}+x$, $\frac{1}{2}-y$, z) (O9···H3 2.427 Å). This may also be a weak C—H···O hydrogen bond. These hydrogen bonds connect the molecules into a sheet parallel to (101). There are no unusually short contacts between the molecules except for the hydrogen bonds.

Experimental

The title compound was recrystallized from an aqueous methanol solution containing 6-hydroxypicolinic acid (enol form), purchased from the Tokyo Kasei Organic Chemical Company.

Crystal data

$C_6H_5NO_3$
$M_r = 139.11$
Monoclinic
$P2_1/n$
$a = 9.346$ (2) Å
$b = 4.5979$ (12) Å
$c = 13.7798$ (14) Å
$\beta = 105.164$ (11)°
$V = 571.5$ (2) Å^3
$Z = 4$
$D_x = 1.617$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 25 reflections
$\theta = 25.46$–29.59°
$\mu = 1.139$ mm^{-1}
$T = 200$ (2) K
Plate
0.30 × 0.12 × 0.03 mm
Orange

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.726$, $T_{\max} = 0.967$
980 measured reflections
935 independent reflections
616 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.030$
$\theta_{\max} = 67.5$°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 5$
$l = -15 \rightarrow 15$
3 standard reflections every 100 reflections intensity variation: +0.39%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.161$
$S = 1.024$
935 reflections
91 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1137P)^2 + 0.0257P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N1	0.4246 (3)	0.7055 (6)	0.57506 (17)	0.0231 (6)
C2	0.3288 (3)	0.6994 (7)	0.4825 (2)	0.0253 (7)
O7	0.3524 (2)	0.8560 (5)	0.41270 (14)	0.0279 (6)
C3	0.2060 (3)	0.5054 (7)	0.4687 (2)	0.0286 (8)
C4	0.1861 (3)	0.3447 (8)	0.5474 (2)	0.0313 (8)
C5	0.2875 (4)	0.3617 (8)	0.6422 (2)	0.0301 (8)
C6	0.4068 (3)	0.5412 (7)	0.6545 (2)	0.0255 (7)
C8	0.5229 (3)	0.5510 (7)	0.7519 (2)	0.0283 (8)
O9	0.5009 (3)	0.4483 (6)	0.82752 (15)	0.0385 (7)
O10	0.6464 (2)	0.6759 (6)	0.74561 (14)	0.0317 (6)

Table 2. *Selected geometric parameters (Å, °)*

N1—H1	0.864	C4—C5	1.401 (4)
N1—C2	1.354 (4)	C5—C6	1.362 (5)
N1—C6	1.376 (4)	C6—C8	1.489 (4)
C2—O7	1.266 (4)	C8—O9	1.210 (4)
C2—C3	1.426 (4)	C8—O10	1.312 (4)
C3—C4	1.365 (5)		
C2—N1—C6	123.5 (3)	C5—C6—N1	119.9 (3)
O7—C2—N1	120.0 (3)	C5—C6—C8	120.7 (3)
O7—C2—C3	123.3 (3)	N1—C6—C8	119.3 (3)
N1—C2—C3	116.7 (3)	O9—C8—O10	125.4 (3)
C4—C3—C2	120.3 (3)	O9—C8—C6	121.1 (3)
C3—C4—C5	120.8 (3)	O10—C8—C6	113.6 (3)
C6—C5—C4	118.8 (3)		

The completeness of the data to a $\theta_{\max}$ value of 67.5° was 90.5% because of the low-temperature equipment. The title structure was solved by direct methods and refined by full-matrix least squares on F^2. All H atoms were located on the difference Fourier map and their positions were fixed. Their isotropic displacement parameters were fixed at $1.2U_{\rm iso}$ of the bound C or N atom, or at $1.5U_{\rm iso}$ of the bound O atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1993). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97

(Sheldrick, 1997). Molecular graphics: *ORTEP*III (Burnett & Johnson, 1996). Software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1286). Services for accessing these data are described at the back of the journal.

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4-Chloro-2-{4-[*p*-(methylsulfonyl)phenyl]-2-oxo-5-phenyl-2,3-dihydro-1,3-oxazol-3-yl}-phenyl Acetate

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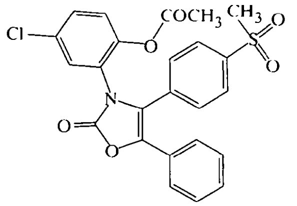
Abstract

In the title triaryloxazolone derivative, $C_{24}H_{18}ClNO_6S$, the three phenyl rings are oriented in a propeller-like fashion around the central oxazolone ring, with dihedral angles of 55.7 (1) (acetoxyphenyl), 56.4 (1) (methylsulfonylphenyl) and 25.0 (1)° (phenyl).

Comment

In the course of investigating the structure–activity relationships of COX-2 (cyclooxygenase II) inhibitors (Thérien *et al.*, 1997), we isolated an unknown product from the condensation reaction of 2-amino-5-chlorobenzoxazole with 2-bromo-2-[4-(methylsulfonyl)-phenyl]-1-phenylethanone. Conversion of this compound to an acetate, (I), permitted crystallization. The subsequent crystal structure determination unequivocally established the connectivity of this compound. Knowing the structure and the results of isotopic labelling studies permitted the proposal of a mechanism for the formation of this triaryloxazolone derivative (Roy *et al.*, 1997).



(I)

The conformation adopted by the molecule is shown in Fig. 1, which also shows the crystallographic numbering scheme. The dihedral angles between the oxazolone ring and the substituent phenyl rings are: 55.7 (1)° with ring C2–C7, 25.0 (1)° with ring C13–C18 and 56.4 (1)° with ring C19–C24. All bond distances and angles agree well with values for similar types of bonds reported in the Cambridge Structural Database (Allen & Kennard, 1993).

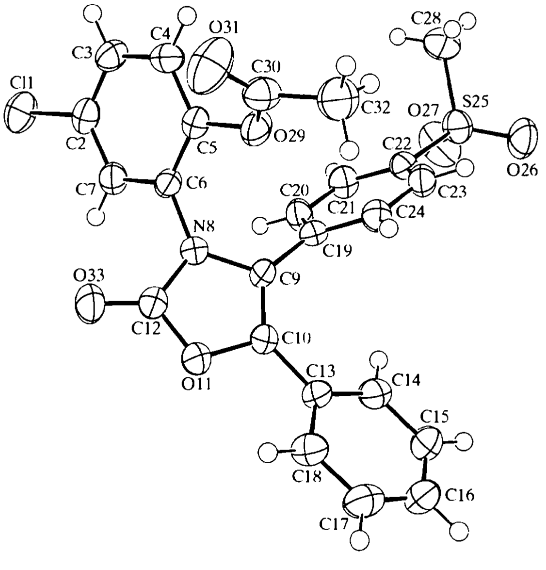


Fig. 1. The molecular conformation of (I) showing the crystallographic numbering scheme. Ellipsoids are drawn at the 50% probability level, while H atoms are represented by circles of arbitrary size.

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