

C(2)—C(1)—C(6)	116.6 (8)	C(5)—C(4)—C(7)	120.4 (10)
C(2)—C(1)—B(1)	122.6 (10)	C(1)—C(6)—C(5)	120.6 (9)
C(6)—C(1)—B(1)	120.7 (10)	C(4)—C(7)—C(8)	125.8 (12)
C(1)—C(2)—C(3)	123.3 (9)	O(1)—B(1)—O(2)	116.9 (9)
C(2)—C(3)—C(4)	119.9 (9)	O(1)—B(1)—C(1)	123.6 (13)
C(3)—C(4)—C(5)	116.9 (8)	O(2)—B(1)—C(1)	119.5 (13)
C(3)—C(4)—C(7)	122.7 (10)		
B(1)—C(1)—C(2)—C(3)	-177.9 (12)	C(2)—C(1)—B(1)—O(2)	24.1 (5)
C(2)—C(1)—B(1)—O(1)	-155.6 (14)	C(6)—C(1)—B(1)—O(2)	-152.9 (14)
C(6)—C(1)—B(1)—O(1)	27.5 (6)		

Direct methods were used to solve the structure. H atoms and phenyl C atoms [C(1)—C(6)] were refined with isotropic displacement parameters. Two grouped displacement parameters for H atoms were employed: (i) for those attached to O atoms; (ii) for those attached to C atoms.

Data collection: *Nicolet R3m Software* (Siemens, 1983). Cell refinement: *Nicolet R3m Software*. Data reduction: *Nicolet R3m Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *NRC-VAX* (Larson *et al.*, 1991). Molecular graphics: *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1036). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

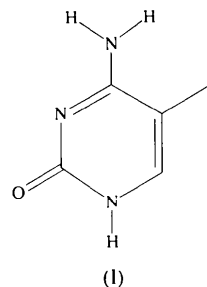
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difference was found between the two molecules. The molecules are held together by N—H···O and N—H···N hydrogen bonds.

Comment

The crystal structures of 5-fluorocytosine (Louis, Low & Tollin, 1982) and 5-bromocytosine (Kato, Takenaka & Sasada, 1979) have been determined as the fundamental compounds for the investigation of the halogenation effect on the base moiety and the interaction between nucleic acids and proteins. Recently, it has been reported that cytosine deaminase can convert the antifungal agent 5-fluorocytosine into the antitumor agent 5-fluorouracil. This suggests importance of halogenated cytosines as medicinal substances (Wallace *et al.*, 1994). Thus, it seemed important to determine the structure of 5-iodocytosine, (I), one of the halogenated derivatives of cytosine.



The molecular packing is stabilized by intermolecular hydrogen bonds: N(1)—H(1)···N(3')(x, 1+y, -1+z) 2.82 (1) Å, N(1')—H(1')···N(3)(1/2+x, -3/2+y, 1+z) 2.83 (1) Å, N(4)—H(4)···O(2')(-1/2+x, 3/2+y, -1+z) 2.90 (1) Å and N(4')—H(4')···O(2)(x, -1+y, 1+z) 2.91 (1) Å.

Note added in proof. Examination of the asymmetric set of atomic coordinates using *Xtal-GX* (Hall, 1995) indicated the presence of a screw-like relationship, although (at the time of going to press) it is uncertain whether this has space-group implications or is non-crystallographic.

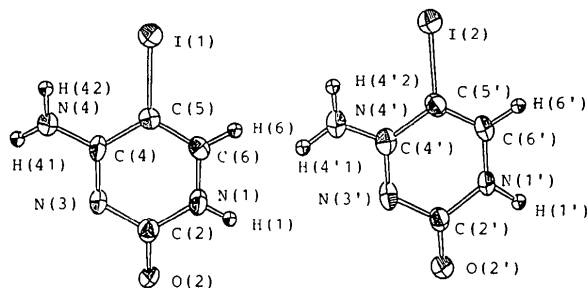


Fig. 1. *ORTEPII* (Johnson, 1976) drawing of the title compound with the atomic numbering scheme, viewed along the *b* axis. Ellipsoids for non-H atoms correspond to 50% probability.

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5-Iodocytosine

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(Received 23 May 1995; accepted 28 June 1995)

Abstract

There are two independent molecules in the asymmetric unit of the title compound, 4-amino-5-iodo-2(1*H*)-pyrimidinone, C₈H₉IN₃O. No significant conformational

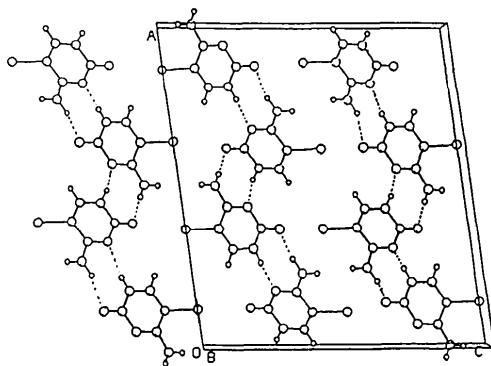


Fig. 2. Packing diagram of the title compound along the *b* axis of the unit cell; intermolecular hydrogen bonds are represented by dashed lines.

Experimental

Single plate crystals were obtained by slow evaporation of a 50% ethanol solution.

Crystal data

$C_4H_4IN_3O$
 $M_r = 237.00$
 Monoclinic
 C2
 $a = 18.248(1) \text{ \AA}$
 $b = 4.442(3) \text{ \AA}$
 $c = 15.913(2) \text{ \AA}$
 $\beta = 98.366(6)^\circ$
 $V = 1276.0(8) \text{ \AA}^3$
 $Z = 8$
 $D_x = 2.467 \text{ Mg m}^{-3}$

Data collection

Rigaku AFC-5R diffractometer
 ω - 2θ scans
 Absorption correction:
 ψ scan (North, Phillips & Mathews, 1968)
 $T_{\min} = 0.85$, $T_{\max} = 1.00$
 1710 measured reflections
 1658 independent reflections
 1441 observed reflections
 $[I > 1.5\sigma(I)]$

Refinement

Refinement on F
 $R = 0.040$
 $wR = 0.045$
 $S = 1.75$
 1441 reflections
 162 parameters
 H-atom parameters not refined
 $w = 4F_o^2/\sigma^2(F_o^2)$

Mo $K\alpha$ radiation
 $\lambda = 0.71069 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 22.25$ – 24.55°
 $\mu = 4.881 \text{ mm}^{-1}$
 $T = 296 \text{ K}$
 Plate
 $0.30 \times 0.10 \times 0.05 \text{ mm}$
 Colorless

$R_{\text{int}} = 0.050$
 $\theta_{\text{max}} = 27.50^\circ$
 $h = 0 \rightarrow 23$
 $k = 0 \rightarrow 5$
 $l = -20 \rightarrow 19$
 3 standard reflections monitored every 150 reflections
 intensity decay: 0.04%

$(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 1.78 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -1.56 \text{ e \AA}^{-3}$
 Extinction correction: none
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$B_{\text{eq}} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
I(1)	0.13396 (4)	0.3887	0.00088 (4)	3.21 (3)
I(2)	0.38614 (3)	0.3542 (3)	0.48718 (4)	3.08 (3)
O(2)	0.1289 (4)	1.173 (2)	-0.3149 (5)	3.1 (3)
O(2')	0.3806 (4)	-0.342 (2)	0.8142 (5)	3.3 (4)
N(1)	0.1903 (4)	0.848 (3)	-0.2183 (5)	2.7 (3)
N(1')	0.4438 (4)	-0.173 (3)	0.7105 (5)	2.6 (4)
N(3)	0.0643 (4)	0.960 (2)	-0.2186 (5)	2.5 (4)
N(3')	0.3219 (4)	-0.014 (2)	0.7164 (6)	2.6 (4)
N(4)	0.0009 (5)	0.758 (3)	-0.1203 (6)	3.3 (4)
N(4')	0.2658 (4)	0.321 (3)	0.6202 (6)	3.5 (4)
C(2)	0.1278 (5)	1.006 (3)	-0.2544 (7)	2.4 (4)
C(2')	0.3818 (5)	-0.181 (3)	0.7518 (6)	2.3 (4)
C(4)	0.0640 (5)	0.786 (3)	-0.1517 (7)	2.6 (4)
C(4')	0.3240 (6)	0.154 (2)	0.6483 (7)	2.3 (4)
C(5)	0.1301 (6)	0.629 (3)	-0.1125 (7)	2.5 (4)
C(5')	0.3887 (6)	0.151 (3)	0.6041 (7)	2.6 (4)
C(6)	0.1923 (5)	0.672 (3)	-0.1505 (6)	2.3 (4)
C(6')	0.4458 (5)	-0.022 (3)	0.6400 (7)	2.8 (4)

Table 2. Selected geometric parameters (\AA , $^\circ$)

I(1)—C(5)	2.09 (1)	N(3)—C(4)	1.32 (1)
I(2)—C(5')	2.06 (1)	N(3')—C(2')	1.37 (1)
O(2')—C(2')	1.22 (1)	N(3')—C(4')	1.32 (1)
O(2)—C(2)	1.22 (1)	N(4)—C(4)	1.33 (1)
N(1)—C(2)	1.39 (1)	N(4')—C(4')	1.32 (1)
N(1)—C(6)	1.33 (1)	C(4)—C(5)	1.45 (1)
N(1')—C(2')	1.39 (1)	C(4')—C(5')	1.46 (1)
N(1')—C(6')	1.31 (1)	C(5)—C(6)	1.37 (1)
N(3)—C(2)	1.38 (1)	C(5')—C(6')	1.35 (2)
C(2)—N(1)—C(6)	123.8 (8)	N(4)—C(4)—C(5)	121 (1)
C(2')—N(1')—C(6')	122.8 (9)	N(3')—C(4')—N(4')	118.6 (9)
C(2)—N(3)—C(4)	121.5 (8)	N(3')—C(4')—C(5')	121 (1)
C(2')—N(3')—C(4')	121.8 (9)	N(4')—C(4')—C(5')	121 (1)
O(2)—C(2)—N(1)	121.8 (9)	I(1)—C(5)—C(4)	122.4 (7)
O(2)—C(2)—N(3)	121.9 (9)	I(1)—C(5)—C(6)	121.6 (8)
N(1)—C(2)—N(3)	116 (1)	C(4)—C(5)—C(6)	116 (1)
O(2')—C(2')—N(1')	120.6 (9)	I(2)—C(5')—C(4')	121.1 (8)
O(2')—C(2')—N(3')	122.7 (9)	I(2)—C(5')—C(6')	123.0 (8)
N(1')—C(2')—N(3')	117 (1)	C(4')—C(5')—C(6')	115 (1)
N(3)—C(4)—N(4)	117.6 (9)	N(1)—C(6)—C(5)	120.7 (9)
N(3)—C(4)—C(5)	121.8 (8)	N(1')—C(6')—C(5')	122.5 (9)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELX86* (Sheldrick, 1985), *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1199). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(Z)-2,3-Bis(3,4-dimethoxyphenyl)propenoic Acid

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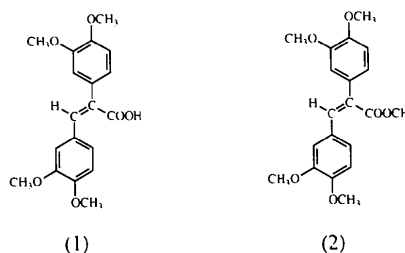
Abstract

The crystal structure determination demonstrates unambiguously that the title compound, C₁₉H₂₀O₆, has the Z configuration. The compound was synthesized starting from 2,3-bis(3,4-dimethoxyphenyl)-2-hydroxypropanoic acid, which was in turn obtained by an alkaline treatment of *trans*-1,3-bis(3,4-dimethoxyphenyl)-2,3-epoxy-1-propanone. ¹H NMR data for the title compound and its methyl ester are reported.

Comment

α -Arylcinnamic acids are of interest as intermediates in the syntheses of stilbenes. Gierer, Lenic, Norén & Szabo-Lin (1974) prepared a series of lignin-related stilbenes using α -arylcinnamic acids as starting materials. Such compounds (and their esters) can also be used as starting materials for the preparation of α -arylcinnamyl alcohols. Certain compounds of this latter type are intermediates in the acid-catalysed reactions of lignin models of the 1,2-diaryl-1,3-propanediol type (Li & Lundquist,

1995). The X-ray work presented in this study establishes the structure and stereochemistry of such an α -arylcinnamic acid, namely, (Z)-2,3-bis(3,4-dimethoxyphenyl)propenoic acid, (1). Since the synthesis of α -arylcinnamyl alcohols from α -arylcinnamic acids usually proceeds *via* the ester derivatives, we also prepared the methyl ester of (1), *i.e.* (2), and examined it by X-ray crystallographic methods. Attempts to obtain good crystals of (2) failed (the crystals often suddenly cracked, a possible explanation being internal strain). However, a structure analysis was performed and it could be concluded that (2) had the expected Z configuration.



The title acid (1), contaminated with minor amounts of the *E* form, was obtained by heating 2,3-bis(3,4-dimethoxyphenyl)-2-hydroxypropanoic acid with acetic

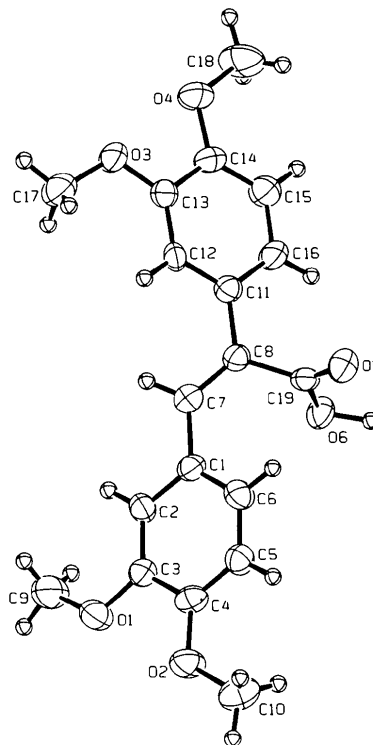


Fig. 1. A perspective drawing (*ORTEPII*; Johnson, 1976) of compound (1), with displacement ellipsoids at the 50% level, showing the numbering of the non-H atoms. H atoms are shown as small spheres of arbitrary radii.