1293 reflections
84 parameters
H-atom parameters not refined
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

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

	x	y	z	$U_{\rm eq}$
S1†	0.54750 (4)	0.24220 (2)	0.81470 (1)	0.0682 (3)
C2	0.70800 (1)	0.23220 (9)	0.73190 (4)	0.0680 (1)
C3	0.73520 (1)	0.15510 (2)	0.64840 (4)	0.0700 (1)
C4†	0.62370 (1)	0.09620 (7)	0.65330 (3)	0.0839 (8)
C5	0.51110 (1)	0.14230 (7)	0.74560 (3)	0.0519 (7)
C6	0.38400 (1)	0.11030 (7)	0.79260 (4)	0.0564 (8)
C7	0.27890 (1)	0.08220 (8)	0.83780 (4)	0.0600 (9)
C8	0.16000 (1)	0.04850 (8)	0.89910 (4)	0.0625 (9)
C9	0.05800 (1)	0.01790 (8)	0.96390 (4)	0.0636 (9)
S4‡	0.62370 (1)	0.09620 (7)	0.65330 (3)	0.0839 (8)
C1‡	0.54750 (4)	0.24220 (2)	0.81470 (1)	0.0682 (3)

† Site occupancy = 0.874. ‡ Site occupancy = 0.126.

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

S1—C5	1.706 (2)	C7—C6	1.189 (2)
S1—C2	1.697 (2)	C8—C9	1.203 (2)
C4—C5	1.454 (2)	C9—C9^{i}	1.373 (2)
C4—C3	1.484 (2)	C3—C2	1.356 (3)
C5—C6	1.417 (2)	C3—S4	1.484 (2)
C5—S4	1.454 (2)	C2—C1	1.697 (2)
C7—C8	1.370 (2)		
C5—S1—C2	93.4 (1)	C8—C7—C6	178.1 (2)
C5—C4—C3	105.8 (2)	C7—C8—C9	177.7 (2)
S1—C5—C4	113.2 (1)	C8—C9—C9^{i}	179.3 (2)
S1—C5—C6	121.0 (1)	C5—C1—C2	93.4 (1)
S1—C5—S4	113.2 (1)		
C2—S1—C5—C4	−0.1 (1)	C3—C4—C5—S1	−0.5 (1)
C2—S1—C5—C6	177.3 (2)	C3—C4—C5—C6	−177.8 (2)
C5—S1—C2—C3	0.8 (2)	C5—C4—C3—C2	1.1 (2)

Symmetry code: (i) $-x, -y, 2 - z$.

The disorder model, with S1 and C4 exchanging their positions in the thiophene ring, was treated by refining the model to include these two atoms having the coordinates of their counterparts, C1 and S4, respectively. After refining, the occupancies of the above atoms were obtained and the extent of disorder was known. The occupancies were then fixed at 0.874 and 0.126 in the final cycles. As suitable ψ-scan data were not available, *DIFABS* (Walker & Stuart, 1983) was used for the absorption correction. The U^{ij} values and their s.u.'s may not be reliable.

Data collection: *CRYSTAN-GM* (Mackay *et al.*, 1994). Cell refinement: *CRYSTAN-GM*. Data reduction: *CRYSTAN-GM*. Program(s) used to solve structure: *SIR* (Altomare *et al.*, 1994). Program(s) used to refine structure: *SIR*. Molecular graphics: *CRYSTAN-GM*. Software used to prepare material for publication: *CRYSTAN-GM*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1072). Services for accessing these data are described at the back of the journal.

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2-Chloro-3-cyano-6-phenylpyridine

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Abstract

The molecular structure of the title compound (2-chloro-6-phenylpyridine-3-carbonitrile, $C_{12}H_7ClN_2$) is presented. The molecule is nearly planar. Despite strong substituent interactions, the aromaticity of the pyridine fragment is very high.

Comment

2,3,6-Substituted pyridines are very useful compounds for the synthesis of natural products with biological activity, such as vitamins (Perez-Medina *et al.*, 1947). Some of them possess bacteriostatic properties (Lesher & Grvet, 1973). The title compound, (I), crystallizes in the $P2_1/c$ space group. The molecular parameters are in good agreement with those of 2-chloro-3-cyano-4-trifluoromethyl-6-phenylpyridine (Guttsait *et al.*, 1987) and with those of α-phenylpyridine derivatives [16 precisely measured derivatives taken from the Cambridge Structural Database (Allen *et al.*, 1979, 1991)]. The only significant differences in the latter case

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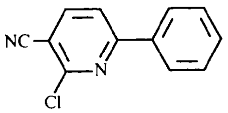
are in the N1—C2 bond length [mean value of 1.342 Å compared with 1.311 (2) Å in (I)] and the bond angles at the substitution positions (N1—C2—C3 and C2—C3—C4).



(I)

The molecule is nearly planar and the angle between the rings is 3.13 (6)°, which is similar to the value of 5° obtained for the two independent molecules of 2-chloro-3-cyano-4-trifluoromethyl-6-phenylpyridine (Guttsait *et al.*, 1987). In other derivatives of α-phenylpyridine [10 unsubstituted in the position *ortho* to the pyridine–phenyl bond retrieved from the Cambridge Structural Database (Allen *et al.*, 1979, 1991)], the angle between the rings varies to a large extent (range 5.1–40.8°, mean 22.2°). Despite strong substituent interactions, the aromaticity (Kruszewski & Krygowski, 1972; Krygowski & Cyrański, 1996*a*) of the pyridine ring is very high (0.977), with a value similar to those observed for other pyridine derivatives (Krygowski & Cyrański, 1996*b*), as well as for 1,2,5-trisubstituted benzene derivatives (Cyrański & Krygowski, 1996). The atomic numbering is given in Fig. 1, while selected bond lengths are given in Table 1.

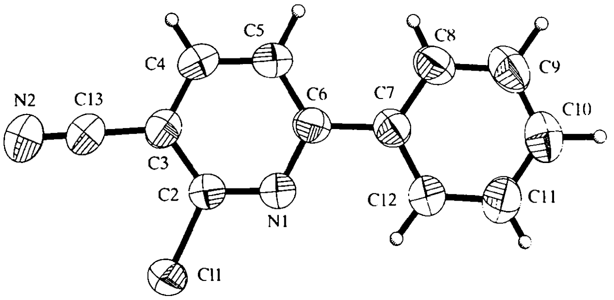


Fig. 1. A view of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

For the synthesis of (I), acetophenone was condensed with ethyl formate in the presence of sodium hydride. The sodium enol salt was treated with cyanoacetamide and 6-phenyl-3-cyano-2-pyridone was formed. Reaction of this pyridone with phosphorous pentachloride gave the title compound, (I), with the same melting point as reported in the literature (Perez-Medina *et al.*, 1947). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{12}H_7ClN_2$
$M_r = 214.65$
Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Monoclinic
$P2_1/c$
$a = 6.585 (1)$ Å
$b = 11.066 (2)$ Å
$c = 14.207 (3)$ Å
$\beta = 101.45 (3)^\circ$
$V = 1014.7 (3)$ Å^3
$Z = 4$
$D_x = 1.405$ Mg m^{-3}
D_m not measured
Cell parameters from 82 reflections
$\theta = 0.2$–79.0°
$\mu = 3.023$ mm^{-1}
$T = 293 (2)$ K
Prism
0.30 × 0.25 × 0.25 mm
Colorless

Data collection

Kuma KM-4 diffractometer
ω/θ scans
Absorption correction: none
3028 measured reflections
2119 independent reflections
1617 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.037$
$\theta_{\rm max} = 79.09^\circ$
$h = -8 \rightarrow 1$
$k = -1 \rightarrow 14$
$l = -17 \rightarrow 17$
3 standard reflections every 100 reflections intensity decay: 1.5%

Refinement

Refinement on F^2
$R(F) = 0.050$
$wR(F^2) = 0.132$
$S = 1.077$
2104 reflections
165 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.1009P)^2 + 0.0744P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.027$
$\Delta\rho_{\rm max} = 0.327$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.386$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.040 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N1—C2	1.311 (2)	C3—C4	1.389 (3)
N1—C6	1.348 (2)	C4—C5	1.373 (3)
C2—C3	1.395 (2)	C5—C6	1.393 (3)
C2—N1—C6	118.4 (2)	C5—C4—C3	119.3 (2)
N1—C2—C3	124.9 (2)	C4—C5—C6	120.1 (2)
C4—C3—C2	116.5 (2)	N1—C6—C5	120.7 (2)
N1—C6—C7—C12	−0.3 (2)	N1—C6—C7—C8	−178.8 (2)
C5—C6—C7—C12	179.2 (2)	C5—C6—C7—C8	0.7 (3)

H atoms were located from a difference map and refined isotropically.

Data collection: *KM*-4 *Software* (Kuma Diffraction, 1991). Cell refinement: *KM*-4 *Software*. Data reduction: *KM*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL*/*PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV1021). Services for accessing these data are described at the back of the journal.

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O—H···O, C—H···O and C—H···$\pi_{\rm arene}$ Intermolecular Interactions in (2*R*/2*S*)-2-(1-Oxo-1,3-dihydroisoindol-2-yl)-3-phenylpropanoic Acid

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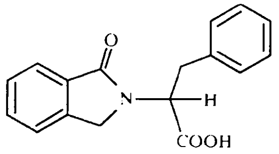
Abstract

The title compound, $C_{17}H_{15}NO_3$, forms a hydrogen-bonded network in the solid state consisting of O—H···O=C, $C_{\rm arene}$—H···O=C and $C_{\rm arene}$—H···$\pi_{\rm arene}$ intermolecular interactions, with shortest O···O, C···O and C···C distances of 2.625 (2), 3.281 (3) and 3.652 (3) Å, respectively. The interplanar angle between the five- and six-membered rings of the isoindole system is 1.07 (14)°, with the carbonyl O atom 0.110 (3) Å from the C_4N ring plane.

Comment

The study of biologically active molecules is of primary importance in medicinal chemistry. Processes such as hormone processing, viral replication and cancer cell invasion are critically dependent on protease enzymes which have recently become attractive target molecules in drug design (Testa *et al.*, 1993). Many inhibitors are based on modified amino acids which incorporate the basic structural features determining normal enzyme–substrate interactions. Phthalimidine (isoindolin-1-one) derivatives often display biological activity as potential anti-inflammatory agents and antipsychotics. The majority of structurally determined phthalimidine systems are either *N*-substituted or have a hydroxy substituent at the 3-position (McNab *et al.*, 1997). The title compound, (I), synthesized as a racemic mixture from DL-phenylalanine, is part of an ongoing study of hydrogen-bonding interactions in amino acid derivatives.



(I)

A view of molecule (I) (*S* configuration) with the atomic numbering scheme is given in Fig. 1 and selected dimensions are given in Table 1. The bond lengths and angles in the heterocyclic ring are similar to those reported previously (McNab *et al.*, 1997) and in agreement with expected values (Orpen *et al.*, 1994). The angle between the five- and six-membered rings of the isoindole system is 1.07 (14)° and the maximum deviation from planarity for an atom in either ring plane is 0.021 (1) Å for C3, with the carbonyl O3 atom 0.110 (3) Å from the C_4N ring plane. This ring is almost perpendicular to both the carboxylic acid CO_2 plane [84.74 (15)°] and the 3-phenyl ring plane

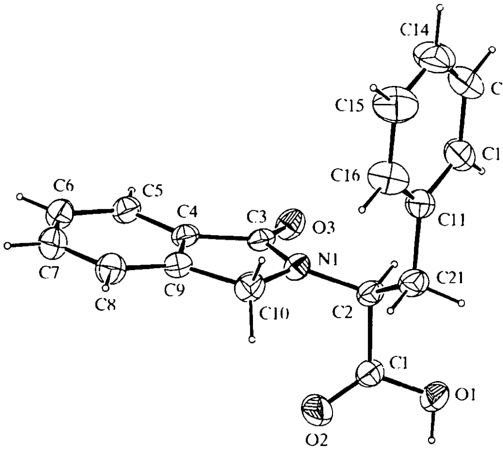


Fig. 1. A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

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