

ORGANIC COMPOUNDS

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N-(2-Hydroxy-5-chlorophenyl)thiophenylacetamide

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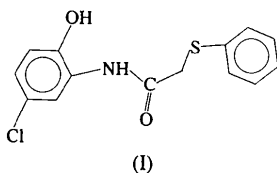
Abstract

In molecules of the title compound, C₁₄H₁₂ClNO₂S, the phenylthio group and the remainder of the heavy-atom skeleton form two different planes which are nearly perpendicular to each other. The molecules are interlinked by O—H···O hydrogen bonds involving the phenolic OH group as donor and the acetamide O atom as acceptor.

Comment

Compounds which contain a benzoxazole ring and their amide derivatives have antibacterial and antifungal (Sadasiwashankar *et al.*, 1985; Elmina *et al.*, 1981; Yalçın *et al.*, 1992), and antitubercular properties (Sycheva *et al.*, 1967); their inhibition of the HIV-1 virus (Carrol *et al.*, 1993) has also been reported. Conney & Burns (1963) and Bray *et al.* (1952) showed that benzoxazole ring systems open during metabolism to give amides. The structures of benzoxazole derivatives and their metabolites are relevant to understanding their biological activity.

Accordingly, the title compound, (I), a metabolite of 5-chloro-2-(phenylthiomethyl)benzoxazole, has been



synthesized and its structure determined. In the molecule, the phenylthio group (Fig. 1) is planar to within 0.0184(6) Å. The rest of the molecule (*i.e.* atoms Cl, O1, O2, N and C1–C8) is planar to within 0.050(2) Å. These two planes are nearly perpendicular [dihedral

angle 92.57(4)°]. Corresponding torsion angles are given in Table 1. The molecules are linked into chains parallel to **a** by O—H···O hydrogen bonds (Table 2). IR and NMR spectra are consistent with hydrogen-bond formation.

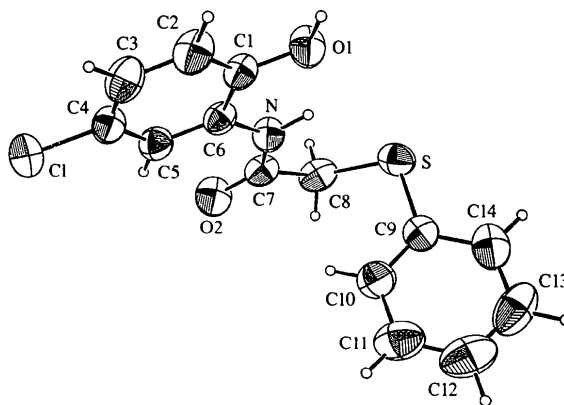


Fig. 1. ORTEP (Fair, 1990) drawing of the C₁₄H₁₂ClNO₂S molecule with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles with arbitrary radii.

Experimental

Thiophenoxyacetic acid (5 mmol, 0.840 g), thionyl chloride (1.5 ml) and benzol (5 ml) were heated under reflux at 353 K for 3 h. Excess reagent and solvent were completely evaporated under reduced pressure. The residue was dissolved in diethyl ether (10 ml) and added dropwise with stirring to a mixture of 4-chloro-2-aminophenol (5 mmol, 0.545 g), NaHCO₃ (10 mmol, 0.840 g), diethyl ether (10 ml) and water (10 ml) at 268 K. The mixture was stirred overnight at room temperature, filtered and the residue sequentially washed with water, 2 N HCl, water and diethyl ether. The residue was crystallized from dichloromethane–hexane. The product was dissolved in hot ethanol, then recrystallized at room temperature (m.p. 441.5–442.0 K).

Crystal data

C₁₄H₁₂ClNO₂S
M_r = 293.76
 Triclinic
*P*1̄
a = 7.471(1) Å
b = 7.735(1) Å
c = 12.330(1) Å
 α = 85.043(7)°
 β = 73.879(8)°
 γ = 82.665(7)°
V = 677.94(14) Å³
Z = 2
D_x = 1.44 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 25 reflections
 θ = 10–18°
 μ = 0.42 mm⁻¹
T = 295 K
 Prismatic
 0.28 × 0.16 × 0.06 mm
 Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer	1974 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	$R_{\text{int}} = 0.008$
Absorption correction: empirical via ψ scans (Fair, 1990)	$\theta_{\text{max}} = 26.3^\circ$
$T_{\text{min}} = 0.973$, $T_{\text{max}} = 0.975$	$h = -8 \rightarrow 8$
2542 measured reflections	$k = -9 \rightarrow 0$
2352 independent reflections	$l = -14 \rightarrow 14$
	3 standard reflections
	frequency: 120 min
	intensity decay: 0.7%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\text{max}} = 0.0002$
$R = 0.033$	$\Delta\rho_{\text{max}} = 0.25 \text{ e } \text{Å}^{-3}$
$wR = 0.043$	$\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{Å}^{-3}$
$S = 1.11$	Extinction correction: none
1974 reflections	Scattering factors from <i>International Tables for X-ray Crystallography</i> (Vol. IV)
172 parameters	
H atoms: see below	
$w = \sigma^2(F) + 0.0004F^2 + 0.50$	

Table 1. Selected geometric parameters (Å , $^\circ$)

Cl—C4	1.745 (2)	O2—C7	1.224 (3)
S—C8	1.792 (2)	N—C6	1.412 (2)
S—C9	1.766 (2)	N—C7	1.335 (2)
O1—C1	1.361 (3)	C7—C8	1.521 (3)
C8—S—C9	103.2 (1)	N—C6—C5	124.4 (2)
C6—N—C7	128.8 (2)	O2—C7—N	124.1 (2)
O1—C1—C2	123.9 (2)	O2—C7—C8	119.5 (2)
O1—C1—C6	116.4 (1)	N—C7—C8	116.4 (2)
Cl—C4—C3	119.5 (2)	S—C8—C7	118.8 (1)
Cl—C4—C5	118.6 (2)	S—C9—C10	123.5 (2)
N—C6—C1	115.6 (2)	S—C9—C14	117.5 (2)
C9—S—C8—C7	80.3 (2)	O2—C7—C8—S	-160.3 (2)
C6—N—C7—C8	178.1 (2)		

Table 2. Hydrogen-bonding geometry (Å , $^\circ$)

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H01 \cdots O2 ¹	0.94	1.74	2.683 (4)	176
C2—H2 \cdots O2 ¹	0.95	2.63	3.304 (5)	128

Symmetry code: (i) $x - 1, y, z$.

All non-H atoms were refined with anisotropic displacement parameters. The H atoms, except H01, H1, H81 and H82, were placed geometrically 0.95 Å from their corresponding C atoms, with $U_{\text{iso}}(\text{H}) = 1.3U_{\text{eq}}(\text{C})$. The H01, H1, H81 and H82 atoms were taken from a difference Fourier map. A riding model was used for all H atoms.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *MolEN* version of *ORTEP*. Software used to prepare material for publication: *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1319). Services for accessing these data are described at the back of the journal.

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4-Nitro-1-(trimethylsilylethynyl)benzene

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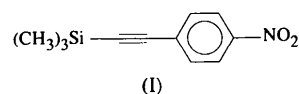
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Abstract

The title molecule, $\text{C}_{11}\text{H}_{13}\text{NO}_2\text{Si}$, lies on a mirror plane, with only one methyl group lying out of plane. The $\text{C}\equiv\text{C}$ triple bond has a length of 1.199 (4) Å. Bond angles $\text{Si}-\text{C}\equiv\text{C}$ and $\text{C}\equiv\text{C}-\text{C}(\text{Ar})$ are 177.9 (3) and 178.0 (3)°, respectively. The $\text{Si}-\text{C}_{\text{sp}^3}$ bond lengths are 1.831 (4) and 1.838 (3) Å, while the $\text{Si}-\text{C}_{\text{sp}}$ distance is 1.839 (3) Å.

Comment

The title compound, (I), was prepared as part of a structural study involving substituted silylethynylbenzene derivatives. The molecule lies on a mirror plane, with



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