Acta Cryst. (1998). C**54**, 488–489

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

Çelik Tarimci,[a] Filiz Ercan,[b] Meltem Koçoğlu[a] and İlkay Ören[c]

[a]*Ankara University, Department of Engineering Physics, Beşevler 06100, Ankara, Turkey,* [b]*Hacettepe University, Department of Engineering Physics, Beytepe 06532, Ankara, Turkey, and* [c]*Ankara University, Faculty of Pharmacy, Department of Pharmaceutical Chemistry, Besevler 06100, Ankara, Turkey. E-mail: filiz@eti.cc.hun.edu.tr*

(*Received 28 January 1997; accepted 4 November 1997*)

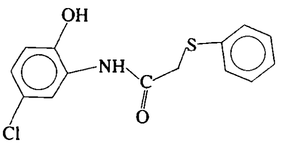
Abstract

In molecules of the title compound, C$_{14}$H$_{12}$ClNO$_2$S, 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 (Sadasivashankar *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.



(I)

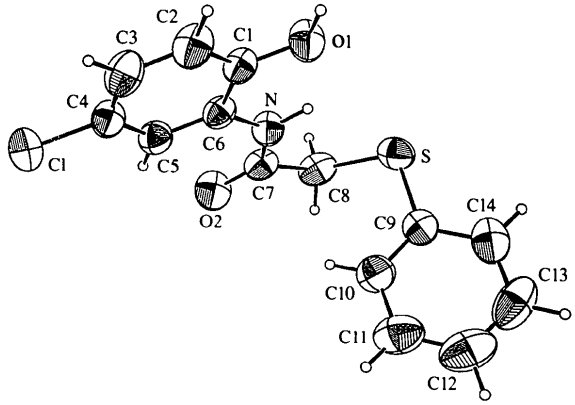


Fig. 1. *ORTEP* (Fair, 1990) drawing of the C$_{14}$H$_{12}$ClNO$_2$S molecule with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles with arbitrary radii.

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), NaHCO$_3$ (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

C$_{14}$H$_{12}$ClNO$_2$S
$M_r = 293.76$
Triclinic
$P\bar{1}$
$a = 7.471\ (1)$ Å
$b = 7.735\ (1)$ Å
$c = 12.330\ (1)$ Å
$\alpha = 85.043\ (7)°$
$\beta = 73.879\ (8)°$
$\gamma = 82.665\ (7)°$
$V = 677.94\ (14)$ Å^3
$Z = 2$
$D_x = 1.44$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10–18°$
$\mu = 0.42$ mm^{-1}
$T = 295$ K
Prismatic
0.28 × 0.16 × 0.06 mm
Yellow

Data collection

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

Refinement

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

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

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, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H01···O2^{i}	0.94	1.74	2.683 (4)	176
C2—H2···O2^{i}	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 $U_{iso}(H) = 1.3U_{eq}(C)$. 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*.

The authors wish to acknowledge the purchase of the CAD-4 diffractometer under Grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey.

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.

References

Bray, H. G., Clowe, R. C. & Thorpe, W. V. (1952). *Biochim. J.* **51**, 70–78.

Carrol, S. S., Olsen, D. B., Bennet, C. D., Gotlib, L., Graham, D. J., Condra, J. H., Stern, A. M., Shafer, J. A. & Kuo, L. C. (1993). *J. Biol. Chem.* **268**, 276–281.

Conney, A. H. & Burns, J. J. (1963). *Ann. N. Y. Acad. Sci.* **86**, 167–177.

Elmina, E. I., Zubair, M. U. & Al-Badr, A. A. (1981). *Antimicrob. Agents Chemother.* **19**, 29–32.

Enraf–Nonius (1993). *CAD-4 EXPRESS*. Version 1.1. Enraf–Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.

Sadasivashankar, M., Reddy, Y. D., Charya, M. A. S. & Reddy, S. M. (1985). *Indian Phytopathol.* **37**, 366–367.

Sycheva, T. P., Kiseleva, I. D. & Shchukina, M. N. (1967). *Khim. Geterotskil. Soedin.* **1**, 43–47.

Yalçın, I., Ören, I., Şener, E., Akın, A. & Uçantürk, N. (1992). *Eur. J. Med. Chem.* **27**, 1–6.

Acta Cryst. (1998). C**54**, 489–491

4-Nitro-1-(trimethylsilylethynyl)benzene

J. Gabriel Garcia,[a]† Bruktawit Asfaw,[a] Augusto Rodriguez[a] and Frank R. Fronczek[b]

[a]*Department of Chemistry, Clark Atlanta University, Atlanta, GA 30314, USA, and* [b]*Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA. E-mail: ggarcia@oberon.cmc.uab.edu*

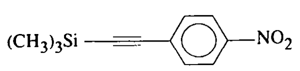
(*Received 25 April 1997; accepted 22 September 1997*)

Abstract

The title molecule, $C_{11}H_{13}NO_2Si$, lies on a mirror plane, with only one methyl group lying out of plane. The C≡C triple bond has a length of 1.199 (4) Å. Bond angles Si—C≡C and C≡C—C(Ar) are 177.9 (3) and 178.0 (3)°, respectively. The Si—C_{sp^3} bond lengths are 1.831 (4) and 1.838 (3) Å, while the Si—C_{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



(I)

[†] Current address: Center for Macromolecular Crystallography, University of Alabama, Birmingham, 262 Basic Health Sciences Building, THT 79, Birmingham, AL 35294-0005, USA.