Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Phenoxymethyl-1,3,4-oxadiazole-2(3*H*)-thione

S. Thamotharan,[a] V. Parthasarathi,[b]* G. Anandha Babu,[b] Raveendra K. Hunnur,[c] Bharati Badami[c] and Anthony Linden[d]

[a]Molecular Biophysics Unit, Indian Institute of Science, Bangalore 560 012, India, [b]School of Physics, Bharathidasan University, Tiruchirappalli 620 024, India, [c]Post-Graduate Department of Studies in Chemistry, Karnatak University, Dharwad 580 003, India, and [d]Institute of Organic Chemistry, University of Zürich, Winterthurerstrasse 190, CH-8057 Zürich, Switzerland

Correspondence e-mail: vpsarati@yahoo.com

Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.006 Å
R factor = 0.047
wR factor = 0.105
Data-to-parameter ratio = 11.9

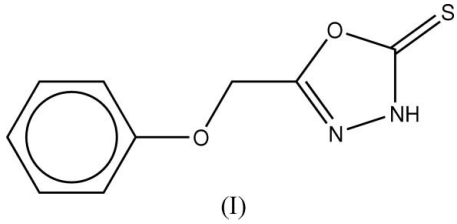
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 10 October 2005
Accepted 14 October 2005
Online 19 October 2005

In the title compound, $C_9H_8N_2O_2S$, the H atom of the thiol group has been transferred to the neighbouring N atom of the oxadiazole ring. Intermolecular N—H···N hydrogen bonds exist between adjacent molecules.

Comment

It is well known that 1,3,4-oxadiazole-2-thione derivatives show a broad spectrum of biological activities (Ram & Vlietinck, 1988; Boschelli *et al.*, 1993). A view of the title compound, (I), with the atomic numbering scheme, is shown in Fig. 1. The dihedral angle between the mean planes of the benzene and 1,3,4-oxadiazole rings is 14.4 (1)°. In (I), the bond lengths and angles are in good agreement with the expected values (Allen *et al.*, 1987). The H atom of the thiol group has been transferred to the adjacent N atom of the oxadiazole ring. The N3—N4 [1.383 (5) Å] and C2═S2 [1.647 (4) Å] bond lengths correspond to the usual single N—N and double C═S distances.



(I)

The crystal structure of (I) is shown in Fig. 2. In the solid state, atom N3 is involved in an intermolecular N—H···N hydrogen bond with atom N4 of the oxadiazole group of an adjacent molecule (Table 1). This hydrogen bond links the molecules into chains, which run parallel to the [010] direction and can be described by a *C*(3) graph-set motif (Bernstein *et al.*, 1995). Atom C6 (*via* H61) acts as a donor for a weak

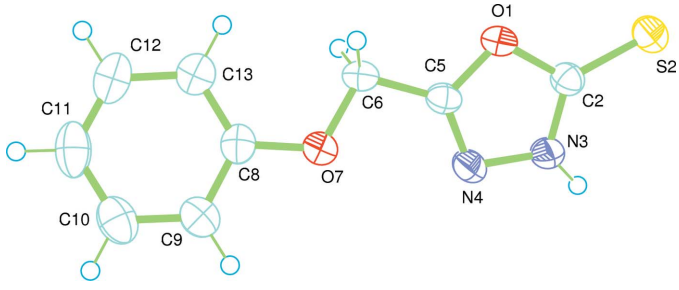


Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radii.

intermolecular C—H···O interaction with atom O1 of a symmetry-related molecule. This weak interaction connects the molecules into chains, which also run parallel to the [010] direction and can be described by a graph-set motif of $C(4)$. In addition, atom C6 (*via* H62) is involved in an intermolecular C—H···π interaction with the benzene ring of a neighbouring molecule [H62···*Cg* = 2.71 Å, C6···*Cg* = 3.459 (4) Å and C6—H61···*Cg* = 132°, where *Cg* is the centroid of the benzene ring at $(x, y-1, z)$].

Experimental

A solution of phenoxyacetic acid hydrazide (0.01 mol) was dissolved in pyridine (10 ml), and carbon disulfide (5 ml) was added with constant stirring. Stirring was continued for 36 h at room temperature. The reaction mixture was then poured into ice-cold water and acidified with dilute HCl. The solid, (I), separated, was filtered off and crystallized from dimethylformamide (m.p. 457–459 K).

Crystal data

$C_9H_8N_2O_2S$
M_r = 208.23
Monoclinic, $P2_1$
a = 9.3233 (8) Å
b = 4.9446 (5) Å
c = 10.2051 (10) Å
β = 91.388 (5)°
V = 470.32 (8) Å^3
Z = 2
D_x = 1.470 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 21914 reflections
θ = 2.0–25.0°
μ = 0.32 mm^{-1}
T = 160 (2) K
Needle, colourless
0.35 × 0.10 × 0.02 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.676, T_{max} = 0.999
6143 measured reflections
1553 independent reflections
1295 reflections with $I > 2\sigma(I)$
R_{int} = 0.083
θ_{max} = 25.0°
$h = -11 \rightarrow 11$
$k = -5 \rightarrow 5$
$l = -11 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.105
S = 1.08
1553 reflections
131 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 0.2737P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack & Bernardinelli (1999, 2000), 636 Friedel pairs
Flack parameter: 0.01 (14)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N4^{i}	0.87 (5)	2.24 (4)	2.899 (5)	132 (3)
C6—H61···O1^{ii}	0.99	2.53	3.416 (4)	148

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z$; (ii) $-x, y+\frac{1}{2}, -z$.

The position of the amine H atom was determined from a difference Fourier map and refined freely along with its isotropic displacement parameter. All remaining H atoms were placed in geometrically idealized positions and were constrained to ride on their parent atoms, with C—H distances in the range 0.95–0.99 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

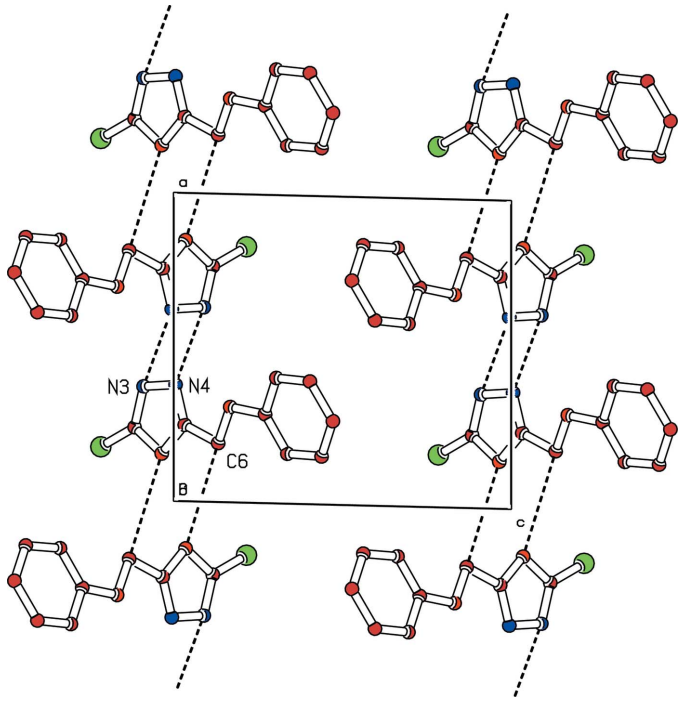


Figure 2
Crystal structure of (I), as projected on to the *ac* plane. N—H···N and C—H···O bonds are indicated by dashed lines. H atoms have been omitted.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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