

5-[3-(Quinolin-8-yloxy)propyl]-1,3,4-oxadiazole-2(3H)-thione

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The title compound, $C_{14}H_{13}N_3O_2S$, crystallizes as a thione tautomer. There are two molecules in the asymmetric unit which differ in conformation. The molecules are connected *via* intermolecular $N-H\cdots N$ and $N-H\cdots O$ hydrogen bonds into a one-dimensional helical structure.

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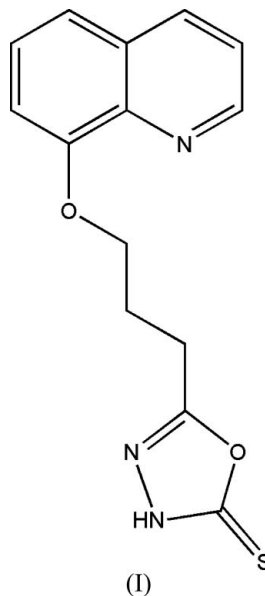
Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
 Mean $\sigma(C-C) = 0.003\text{ \AA}$
 R factor = 0.041
 wR factor = 0.138
 Data-to-parameter ratio = 13.0

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

Comment

In the course of our studies on 1,3,4-oxadiazole-2-thione derivatives the title compound, (I), was synthesized and its crystal structure determined. 1,3,4-Oxadiazole-2-thiones are known to possess a broad spectrum of biological activities (Ram & Vlietinck, 1988; Boschelli *et al.*, 1993; Bahadur & Pandey, 1980).



The asymmetric unit of (I) contains two molecules in the usual thione tautomeric form (Du *et al.*, 2004; Thamotharan *et al.*, 2005) (Table 1). The two molecules differ in conformation. In one molecule, the conformation along the $O3-C24-C25-C26-C27-O4$ bond sequence is *trans-trans-trans*, whereas in the second molecule the conformation is *(+)gauche-trans-(-)gauche* along the corresponding $O1-C10-C11-C12-C13-O2$ bond sequence (Table 1). Despite these differences, the dihedral angles between the mean planes of the 1,3,4-oxadiazole and the quinoline rings are not very different [$11.3(2)$ and $22.6(1)^\circ$ for the molecules containing atoms $O3$ and $O1$, respectively]. The molecules are connected *via* intermolecular $N-H\cdots N$ and $N-H\cdots O$ hydrogen bonds into a one-dimensional helical structure (Table 2 and Fig. 2).

Experimental

To a solution of 4-(quinolin-8-yloxy)butanohydrazide (0.01 mol) in ethanol (30 ml), potassium hydroxide (0.0125 mol) and carbon disulfide were added (0.014 mol). The solution was refluxed for 9 h and the solvent was evaporated under reduced pressure. The residue was dissolved in water and acidified with dilute HCl. The solid (I) which precipitated was filtered off and recrystallized from a mixture of dimethylformamide and ethanol (5:1) (m.p. 505–506 K). Analysis calculated for C₁₄H₁₃N₃O₂S: C 58.52, H 4.56, N 14.62%; found: C 58.50, H 4.58, N 14.61%. Crystals of (I) suitable for single-crystal X-ray analysis were selected directly from the sample after recrystallization.

Crystal data

C₁₄H₁₃N₃O₂S
 M_r = 287.33
 Triclinic, P $\bar{1}$
 a = 7.3208 (5) Å
 b = 11.5523 (7) Å
 c = 16.7934 (10) Å
 α = 84.271 (3)°
 β = 89.326 (4)°
 γ = 75.357 (2)°

V = 1367.13 (15) Å³
 Z = 4
 D_x = 1.396 Mg m⁻³
 Mo Kα radiation
 μ = 0.24 mm⁻¹
 T = 293 (2) K
 Block, colourless
 0.44 × 0.26 × 0.15 mm

Data collection

Bruker SMART diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 T_{min} = 0.901, T_{max} = 0.965

7016 measured reflections
 4781 independent reflections
 4241 reflections with I > 2σ(I)
 R_{int} = 0.015
 θ_{max} = 25.0°

Refinement

Refinement on F²
 R[F² > 2σ(F²)] = 0.041
 wR(F²) = 0.138
 S = 1.00
 4781 reflections
 369 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

w = 1/[σ²(F_o²) + (0.1P)²
 + 0.33P]
 where P = (F_o² + 2F_c²)/3
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.46 e Å⁻³
 Δρ_{min} = -0.32 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

S1–C14	1.6467 (19)	N3–N2	1.388 (2)
S2–C28	1.6543 (19)	N6–N5	1.392 (2)
O2–C13–C12–C11	−71.7 (2)	C25–C26–C27–O4	−168.98 (17)
C13–C12–C11–C10	−171.58 (16)	C27–C26–C25–C24	173.48 (17)
O1–C10–C11–C12	72.6 (2)	O3–C24–C25–C26	−176.73 (17)

Table 2

Hydrogen-bond geometry (Å, °).

D–H...A	D–H	H...A	D...A	D–H...A
N3–H1...N4	0.93 (2)	1.84 (2)	2.770 (2)	174 (2)
N3–H1...O3	0.93 (2)	2.54 (2)	3.036 (2)	113.6 (17)
N6–H29...N1 ⁱ	0.91 (2)	1.89 (2)	2.782 (2)	165 (2)

Symmetry code: (i) x − 1, y, z.

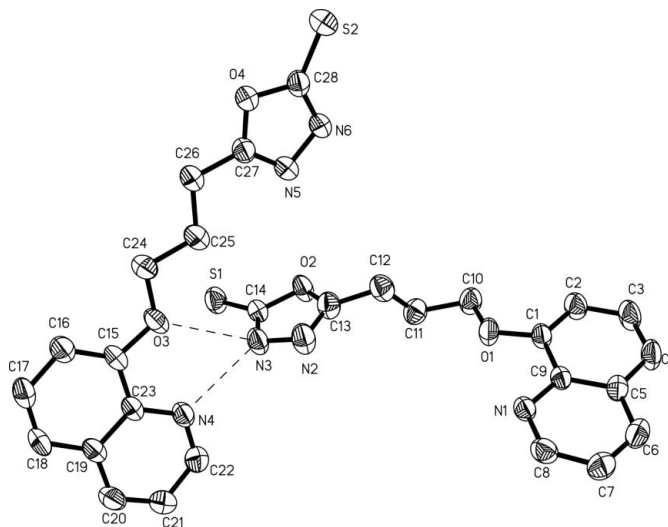


Figure 1

The asymmetric unit of (I) showing the atom labelling and displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity. The dashed lines indicate hydrogen bonds.

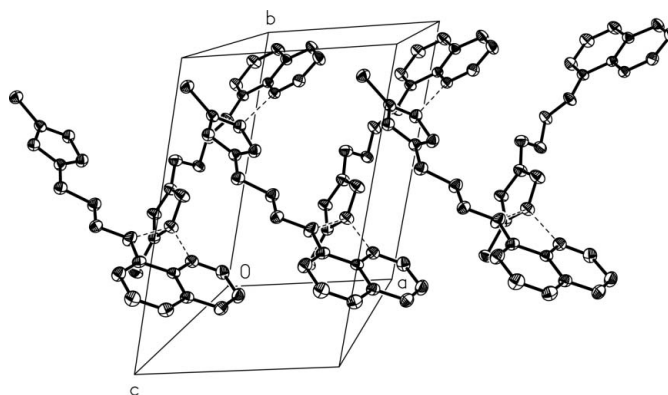


Figure 2

The helical structure formed by hydrogen bonds (dashed lines) in (I). H atoms have been omitted for clarity.

All H atoms were initially located in a difference Fourier map. The methylene H atoms were constrained to an ideal geometry, with C–H = 0.97 Å and U_{iso}(H) = 1.5U_{eq}(C). The positions of the amine H atoms were refined freely along with isotropic displacement parameters. All quinoline H atoms were placed in geometrically idealized positions, with C–H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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