

3-(4-Methylphenoxy)methyl)-4-phenyl-1H-1,2,4-triazole-5(4H)-thione

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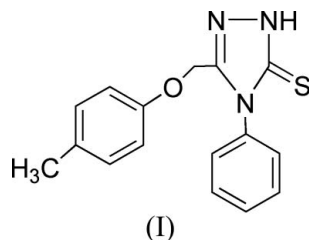
Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(C-C) = 0.003$ Å
 R factor = 0.039
 wR factor = 0.114
Data-to-parameter ratio = 15.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the crystal structure of the title compound, $C_{16}H_{15}N_3OS$, intermolecular $N-H \cdots S$ hydrogen bonds link the molecules together as characteristic dimers, which are further stabilized by weak intermolecular benzene-ring $\pi-\pi$ interactions along the a -axis direction.

Comment

During the last few decades, considerable attention has been devoted to 1,2,4-triazole derivatives for their comprehensive bioactivities, such as antimicrobial (Holla *et al.*, 1998), analgesic (Turan *et al.*, 1999), antitumor (Demirbas *et al.*, 2002), antihypertensive (Paulvannan *et al.*, 2000) and antiviral activities (Kritsanida *et al.*, 2002). The broad biological activities that the 1,2,4-triazoles show may be due to the presence of the $-N-C=S$ unit (Omar *et al.*, 1986). We are interested in the synthesis and biological activities of aryloxyacetyl hydrazide derivatives and report here the synthesis and crystal structure of the title compound, (I).



As shown in Fig. 1, the title compound contains a triazole ring and two benzene rings. The triazole ring, the C4–C9 benzene ring and the C11–C16 benzene ring have mean

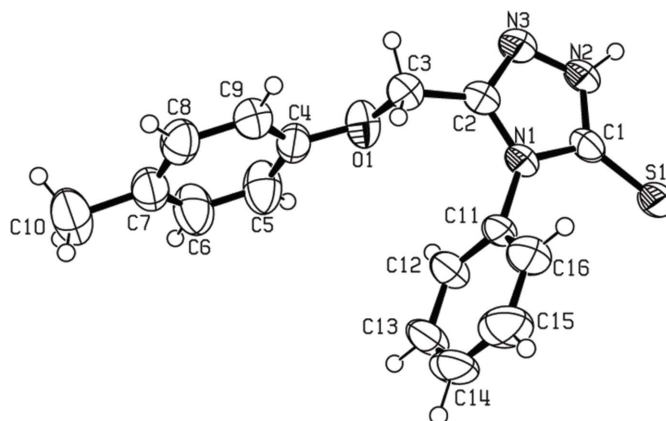


Figure 1

A plot of title compound, with displacement ellipsoids drawn at the 50% probability level.

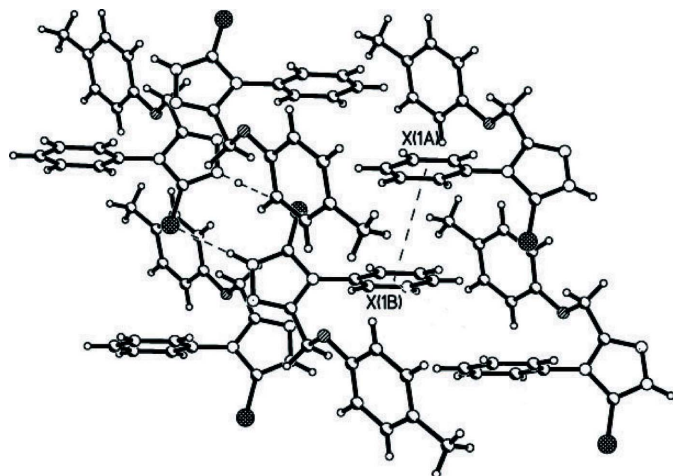


Figure 2

Part of the packing of the title compound. *X(1A)* and *X(1B)* denote the centroids of the C11–C16 benzene rings at (*x*, *y*, *z*) and (1 + *x*, *y*, *z*), respectively.

deviations of 0.0009 (2), 0.0076 (3) and 0.0026 (3) Å, respectively. The dihedral angle between the C11–C16 benzene ring and the triazole ring is 72.8 (3)°.

Hydrogen bonds are observed in the crystal structure (Fig. 2 and Table 1), linking the molecules into centrosymmetric dimers. Weak π – π interactions between the phenyl rings are also observed (Table 2). Dimers are linked through face-to-face π – π interactions; these are responsible for the formation of the solid-state structure (Fig. 2). According to recent calculations (Tsuzuki *et al.*, 2002; Hobza *et al.*, 1996), the slipped-parallel structure of the phenyl dimer shows significant intermolecular π – π interactions. From Fig. 3, it can be seen that the dimers are stabilized by weak π – π interactions along the *a*-axis direction.

Experimental

The synthesis of the title compound was carried out by refluxing a 2 mol l⁻¹ NaOH solution of 1-(4-methylphenoxyacetyl)-4-arylthiosemicarbazide (10 mmol) for 2 h. Colorless single crystals were obtained by slow evaporation of an ethanol solution of the compound over about a week.

Crystal data

C ₁₆ H ₁₅ N ₃ OS	<i>V</i> = 763.8 (6) Å ³
<i>M_r</i> = 297.37	<i>Z</i> = 2
Triclinic, <i>P</i> $\bar{1}$	<i>D_x</i> = 1.293 Mg m ⁻³
<i>a</i> = 7.182 (3) Å	Mo <i>K</i> α radiation
<i>b</i> = 9.846 (4) Å	μ = 0.21 mm ⁻¹
<i>c</i> = 11.644 (5) Å	<i>T</i> = 293 (2) K
α = 71.283 (7)°	Block, colorless
β = 78.783 (7)°	0.52 × 0.47 × 0.25 mm
γ = 89.372 (7)°	

Data collection

Bruker SMART CCD area-detector diffractometer	5977 measured reflections
φ and ω scans	2971 independent reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 1998)	2302 reflections with <i>I</i> > 2σ(<i>I</i>)
<i>T_{min}</i> = 0.897, <i>T_{max}</i> = 0.949	<i>R_{int}</i> = 0.017
	θ_{\max} = 26.0°

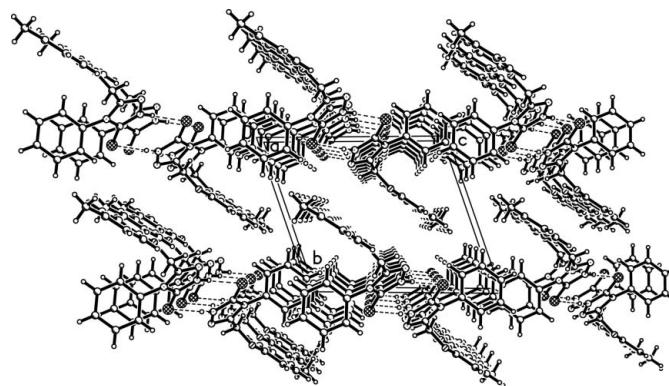


Figure 3

Packing diagram of the title compound.

Refinement

Refinement on *F*²
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.114$
S = 1.03
 2971 reflections
 195 parameters
 H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 0.1617P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.22 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.21 \text{ e } \text{Å}^{-3}$$

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N2–H2...S1 ⁱ	0.88 (2)	2.44 (2)	3.3163 (18)	175.9 (18)

Symmetry code: (i) $-x, -y + 2, -z + 1$.

Table 2

π – π Interactions (face-to-face) (°, Å).

X(1A) and *X(1B)* denote the centroids of the C11–C16 benzene rings shown in Fig. 2.

<i>X(j)</i> → <i>X(k)</i>	Dihedral angle (<i>j</i> , <i>k</i>)	Distance between the centroids
<i>X(1A)</i> → <i>X(1B)</i>	0	4.116

H atoms were positioned geometrically, with C–H distances in the range 0.93–0.97 Å and an N–H distance of 0.88 Å, and refined using a riding model, with *U*_{iso}(H) = 1.5*U*_{eq}(C) for methyl H atoms and 1.2*U*_{eq}(C,N) for the other H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1998); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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