Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-(4-*n*-Butyloxyphenyl)-2-(2-mercaptoethyl)-1,2,4-triazole-3-thione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.055
wR factor = 0.144
Data-to-parameter ratio = 16.2

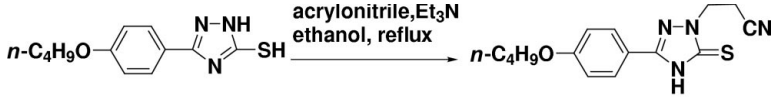
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 June 2004
Accepted 17 June 2004
Online 26 June 2004

In the title molecule, $C_{15}H_{18}N_4OS$, all the bond lengths and angles have normal values. The aromatic triazole ring and the benzene ring are conjugated together.

Comment

The 1,2,4-triazole, (1) (Wang *et al.*, 1997), and related compounds exhibit interesting and useful biological activities (Eisa, 1990). The encouraging biological activities of these heterocycles prompted us to synthesize derivatives of (1), which may be suitable targets for antibiotic design. The title compound, (2), was synthesized by the Michael reaction of a molar ratio of (1) and acrylonitrile in the presence of triethylamine. Since the acidity of NH groups is stronger than that of the SH group on the ring of the triazole, the *N*-propionitrile was produced. The Michael reaction position and the structure of (2) were confirmed by X-ray single-crystal diffraction analysis.



(1) (2)

The crystal structure data show that all the bond lengths and angles have normal values in (2). One molecule of (2) constitutes the asymmetric unit. The N1/C11/N2/N3/C12 ring and atom S1 are approximately cpplanar. The bond length of C12—S1 [1.643 (3) Å] is characterized as a C═S double bond, when compared with the literature value for the length of a C—S single bond [1.731 (2) Å; Tripolt *et al.*, 1993] and the length of a C═S double bond connected to a triazole ring [1.669 (2) Å; Seccombe & Kennard, 1973]. This suggests extensive conjugation of the C═S bond with the triazole ring. The aromatic triazole ring and the C5–C10 benzene ring are conjugated together, with a torsion angle between the triazole and benzene rings of only 2.11 (1)°.

Experimental

To a well stirred solution of 3-(4-*n*-butyloxyphenyl)-5-mercapto-1,2,4-triazole (5 mmol), (1), anhydrous ethanol (5 ml) and triethylamine (5.2 mmol) was added acrylonitrile (5.2 mmol) and the resulting solution refluxed for 30 min (monitored by thin-layer chromatography). At the end of the reaction, the mixture was cooled to room temperature, neutralized and the solvent was removed *in vacuo*. The crude product was purified by column chromatography (silica gel, 20 g; petroleum ether–trichloromethane–ethanol = 5:2:0.5) to give compound (2) as white crystals. White single crystals of (2) suitable for X-ray crystallographic analysis were obtained by recrystallization

from ethyl acetate. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.85–7.82 (*d*, H6, H10), 6.99–6.96 (*d*, H7, H6), 4.43–4.38 (*t*, H13*A*, H13*B*), 4.04–4.00 (*m*, H4*A*, H4*B*), 3.01–2.96 (*m*, H14*A*, H14*B*), 1.82–1.77 (*t*, H3*A*, H3*B*), 1.72–1.59 (*s*, H1), 1.54–1.46 (*t*, H2*A*, H2*B*), 1.01–0.96 (*t*, H1*A*, H1*B*, H1*C*); ^{13}C NMR (300 MHz, $CDCl_3$): δ 176.0, 162.7, 159.9, 128.4, 116.1, 115.1, 113.9, 68.0, 44.6, 31.0, 19.1, 16.0, 13.7.

Crystal data

$C_{15}H_{18}N_4OS$	D_x = 1.279 Mg m^{-3}
M_r = 302.39	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 864 reflections
a = 5.3608 (11) Å	θ = 2.4–18.1°
b = 25.620 (5) Å	μ = 0.21 mm^{-1}
c = 11.554 (2) Å	T = 293 (2) K
β = 98.133 (5)°	Prism, white
V = 1570.9 (5) Å^3	0.3 × 0.2 × 0.2 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3088 independent reflections
	2568 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.027
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	θ_{max} = 26.0°
	$h = -6 \rightarrow 3$
T_{min} = 0.95, T_{max} = 0.96	$k = -31 \rightarrow 31$
8435 measured reflections	$l = -14 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 1.12P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.055	
$wR(F^2)$ = 0.144	
S = 0.93	$(\Delta/\sigma)_{max} < 0.001$
3088 reflections	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
191 parameters	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
H-atom parameters constrained	

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å; N—H = 0.86 Å) and refined as riding, with U_{iso} = 1.2 or 1.5 times U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *MERCURY* (Version 1.2.1; Bruno *et al.*, 2002).

We are grateful to the National Natural Science Foundation of China for financial support (No. 0205B026).

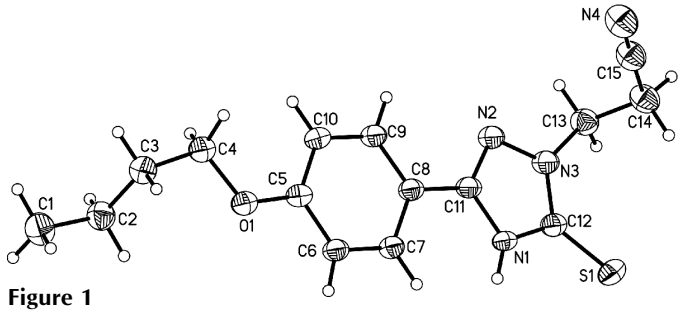


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids.

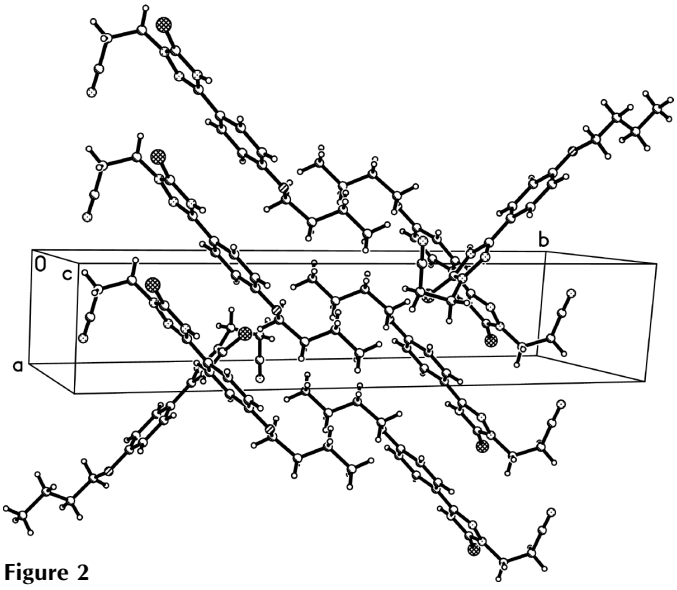


Figure 2
The packing of the title compound, viewed approximately down the *c* axis.

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