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5-(4-Methoxyphenyl)-3-(4-nitrobenzylsulfanyl)-4,5-dihydro-3*H*-1,2,4-triazole

Ghulam Qadeer,[a] Nasim Hasan Rama,[a]* Muhammad Zareef[a] and Xin-Hua Li[b]

[a]Department of Chemistry, Quaid-i-Azam Univeristy, Islamabad 45320, Pakistan, and [b]College of Chemistry and Materials Engineering, Xueyuan Road, Wenzhou University, Zhejiang Wenzhou 325027, People's Republic of China

Correspondence e-mail: nasimhrama@yahoo.com

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.092
wR factor = 0.227
Data-to-parameter ratio = 12.6

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

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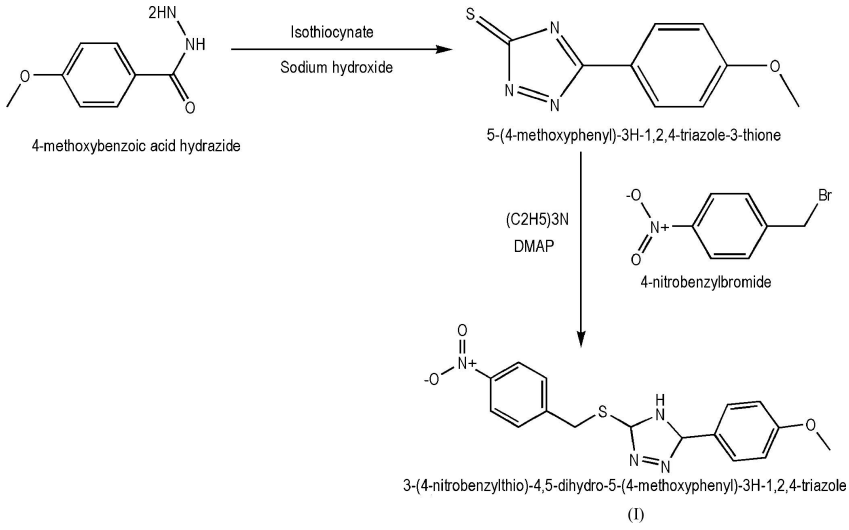
In the title compound, $C_{16}H_{14}N_4O_3S$, the electron delocalization in the triazole ring is reflected in the C—N bond lengths.

Comment

1,2,4-Triazole derivatives possess important pharmacological activities such as antimicrobial (Witkoaski *et al.*, 1972), fungicidal (Heuback *et al.*, 1979), insecticidal (Tanaka, 1974), anticonvulsant (Griffin & Mannion, 1986), antidepressant (Hanna *et al.*, 1988) and antitumor (Jenkin *et al.*, 1989). In a continuation of our interest in the chemical and pharmacological properties of triazole derivatives, we have synthesized a series of new compounds and we report here the structure of the title compound, (I).



(I)

In the molecule of (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). Rings *A* (C2–C7), *B* (N1–N3/C8/C9) and *C* (C11–C16) are, of course, planar and the dihedral angles between them are *A*/*B* = 15.56 (4), *A*/*C* = 69.99 (5) and *B*/*C* = 82.64 (5)°. In ring *B*, the N1—C8 [1.355 (5) Å] and N1—C9 [1.343 (5) Å] bonds are longer than a typical C=N double bond and shorter than a typical C—N single bond, indicating electron delocalization in the ring.

Experimental

A mixture of 4-methoxybenzoic acid hydrazide (1.66 g, 10 mmol) and HNCS (0.6 ml, 10 mmol), which were prepared by standard procedures (Furniss *et al.*, 1978; Gilman & Blatt, 1967), was dissolved in ethanol (180 ml), and refluxed for 7 h. The reaction mixture was cooled to yield the solid thiosemicarbazide. It was filtered, recrystallized from aqueous ethanol (30%), and then dissolved in aqueous sodium hydroxide (4 *N*) and refluxed for 6.5 h. The reaction mixture

was cooled, filtered and acidified with HCl. The solid product obtained, triazole-3-thione, was filtered off, washed with water and recrystallized from ethanol. Triazole-3-thione (1.08 g, 10.8 mmol), Et_3N (0.025 g, 0.25 mmol) and 4-dimethylaminopyridine, (DMAP), (0.025 g, 0.20 mmol) were stirred in $CHCl_3$ (25 ml) for 15 min., and then 4-nitrobenzyl bromide (0.12 g, 0.5 mmol) was added. The reaction mixture was stirred for 9 h at 323–343 K, washed with dilute HCl, brine and water, and then dried over Na_2SO_4 (anhydrous). The excess solvent was distilled off and the product was recrystallized from aqueous ethanol to obtain (I) (yield 73%, m.p. 432–433 K). Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperture.

Crystal data

$C_{16}H_{14}N_4O_3S$
M_r = 342.38
Triclinic, $P\overline{1}$
a = 8.0207 (17) Å
b = 8.1947 (18) Å
c = 12.536 (3) Å
α = 93.926 (4)°
β = 91.031 (4)°
γ = 108.050 (4)°
V = 780.9 (3) Å^3
Z = 2
D_x = 1.456 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 298 (2) K
Block, colorless
0.34 × 0.15 × 0.14 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.948, T_{max} = 0.959
4154 measured reflections
2740 independent reflections
2355 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 25.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.092
$wR(F^2)$ = 0.227
S = 1.17
2740 reflections
218 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0894P)^2 + 1.1058P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

H atoms were positioned geometrically, with N—H = 0.86 Å and C—H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,N), where x = 1.5 for methyl H, and x = 1.2 for all other H atoms.

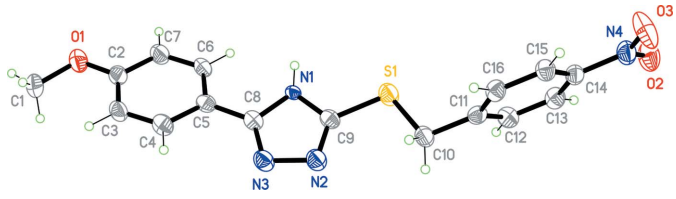


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick,1997); program(s) used to refine structure: *SHELXL97* (Sheldrick,1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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