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4-*tert*-Butyl-5-(2,4,5-trimethoxybenzyl)-thiazol-2-amine

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.119
Data-to-parameter ratio = 17.7

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

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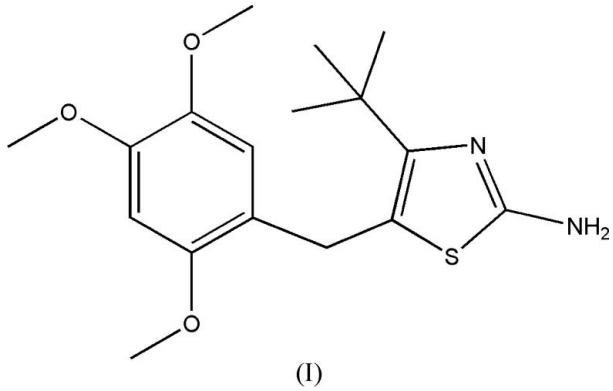
The title compound, $C_{17}H_{24}N_2O_3S$, has been synthesized from 2-bromo-4,4-dimethyl-1-(2,4,5-trimethoxyphenyl)pentan-3-one *via* cyclization by thiourea, acidification by hydrogen bromide which is the by-product of the cyclic reaction, and then neutralization by an aqueous solution of ammonia. Geometric parameters are in the usual ranges. The dihedral angle between the benzene ring and the thiazole ring is 65.9 (2)°. The crystal structure shows N—H···N and N—H···O hydrogen bonds.

Comment

Thiazole derivatives are well known for diverse biological activities and play a key role in antitubercular (Bambas, 1945), antiflammatory (Lombardino *et al.*, 1973), and antibacterial drugs (Talley *et al.*, 1996). It is found that sulfonamide-substituted 4,5-diarylthiazoles in particular have inhibitory activity against COX-2 *in vitro* (Carter *et al.*, 1999). We report here the synthesis and structure of the title compound, (I), which is a new thiazole derivative.



(I)

Fig. 1 shows the molecular structure of (I). The dihedral angle between the benzene ring and the thiazole ring is 65.9 (2)°. The crystal structure is stabilized by N—H···N and N—H···O hydrogen bonds. Two N—H···N hydrogen bonds generate a centrosymmetric $R_2^2(8)$ motif (Bernstein *et al.*, 1995).

Experimental

A solution of thiourea (0.03 mol) and 2-bromo-4,4-dimethyl-1-(2,4,5-trimethoxyphenyl)pentan-3-one (0.03 mol) in ethanol (70 ml) was refluxed for 9 h (monitored by TLC). Part of the solvent was evaporated, and the resulting precipitate was filtered off and dried, giving a yellowish crystalline substance which was the hydrobromide of (I) (m.p. 495.9–496.5 K). The salt was directly dissolved in ethanol

and neutralized with an aqueous solution of ammonia. The precipitate of (I) was filtered off, washed with water and dried (m.p. 427.8–428.2 K). Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.35 (*s*, 9H, $C(CH_3)_3$), 3.78, 3.81, 3.89 (3 × *s*, 9H, 3 × OCH_3), 4.03 (*s*, 2H, CH_2), 4.74 (*br*, 2H, NH_2), 6.52 (*s*, 1H, phenyl 3-H), 6.67 (*s*, 1H, phenyl 5-H). Crystals suitable for X-ray structure determination were obtained by slow evaporation of an ethanol solution at room temperature.

Crystal data

$C_{17}H_{24}N_2O_3S$
M_r = 336.44
Monoclinic, $P2_1/n$
a = 12.9869 (7) Å
b = 7.9512 (4) Å
c = 17.0466 (9) Å
β = 99.070 (1)°
V = 1738.25 (16) Å^3
Z = 4
D_x = 1.286 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 173 (2) K
Block, colorless
0.48 × 0.44 × 0.34 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.909, T_{max} = 0.934
10047 measured reflections
3783 independent reflections
2887 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.119
S = 1.05
3783 reflections
214 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.7859P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N1^{i}	0.88	2.27	3.032 (2)	146
N2—H2B···O2^{ii}	0.88	2.35	3.181 (2)	157
N2—H2B···O3^{ii}	0.88	2.42	3.117 (2)	136

Symmetry codes: (i) $-x+2, -y+1, -z$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

All H atoms were refined using a riding model, with N—H distances of 0.88 and C—H distances of 0.95 (aromatic), 0.98 (methyl) and 0.99Å (methylene H), and with U_{iso}(H)=1.2U_{eq}(C,N) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate but not to tip.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

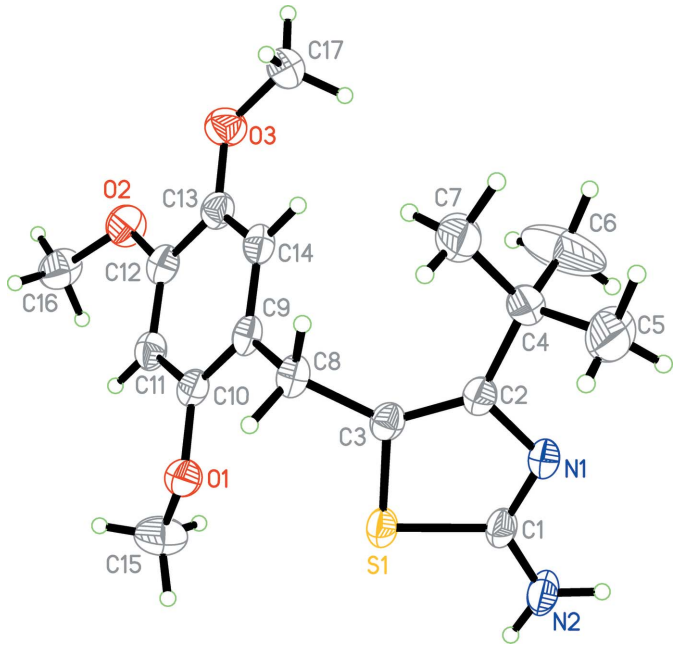


Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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