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4-*tert*-Butyl-1-isopropyl-1*H*-imidazole-2(3*H*)-thione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.061
wR factor = 0.197
Data-to-parameter ratio = 23.3

For 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, $C_{10}H_{18}N_2S$, dimers are formed by N—H···S hydrogen bonding. These dimers are located around centers of inversion.

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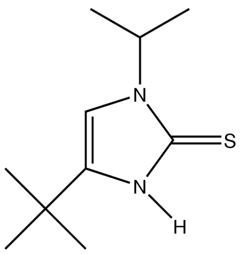
Comment

The title compound, (I), was formed by accident, instead of the sulfenamide which was expected. A search in the Cambridge Structural Database (Version 5.25; Allen, 2002) for the imidazoline-2-thione fragment alkyl- or aryl-substituted at the 1,4-positions, yielded only four structures, namely 1-*p*-tolyl-4-(α-D-*erythro*furanosyl)imidazoline-2-thione (refcode TOEFIM10; Barragán *et al.*, 1977), 1-*p*-tolyl-4-(β-D-*erythro*-idazoline-2-thione (refcode BTEFIM10; Barragán *et al.*, 1978), 1-methyl-4-(β-D-*erythro*furanosyl)imidazoline-2-thione (refcode MEFIZT10; Conde *et al.*, 1975) and 1-*p*-chlorophenyl-4-(α-D-*erythro*furanosyl)-4-imidazoline-2-thione (refcode CEPFIM10; Pérez-Garrido *et al.*, 1974). The C—S bond distance of 1.697 (3) Å in (I) is between the value for a C—S single-bond distance of about 1.81 Å and that for a double-bond distance of about 1.56 Å, and it is in agreement with the C—S distance, 1.698 (2) Å, in imidazoline-2-thione hemihydrate (Raper *et al.*, 1984), as one representative example.



(I)

In the crystal structure of (I), the molecules are linked into centrosymmetric $R_2^2(8)$ dimers (Bernstein *et al.*, 1995) by N—H···S hydrogen bonding. The H···S distance is relatively short and the N—H···S angle is nearly linear (Table 2).

Experimental

All reactions were carried out under an atmosphere of purified nitrogen. Solvents used were dried and distilled prior to use. Bis(4-*tert*-butyl-1-isopropylimidazol-2-yl) disulfide was purchased from Aldrich. In a 1000 ml three-necked flask equipped with an overhead stirrer, silver nitrate (7.8 g, 0.045 mol) was dissolved in methanol (400 ml). After stirring for 1 h at room temperature, an equivalent amount of bis(4-*tert*-butyl-1-isopropyl-2-imidazolyl) disulfide was added and the reaction mixture was cooled in an ice bath. An excess of *N*,*N*-dibenzylamine (0.225 mol) was added, and the reaction

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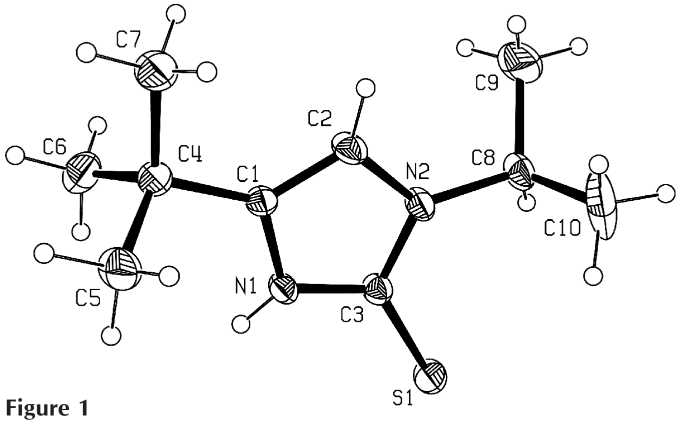

Figure 1
View of the title compound, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

mixture was stirred overnight. The silver mercaptide was removed by filtration and the filtrate was evaporated under reduced pressure at 303–313 K. The residue was extracted with diethyl ether, washed with water (4 × 100 ml) and dried over $MgSO_4$. Single crystals were obtained by slow evaporation of the ether solution.

Crystal data

$C_{10}H_{18}N_2S$
$M_r = 198.32$
Monoclinic, $P2_1/c$
$a = 10.051$ (2) Å
$b = 10.176$ (2) Å
$c = 11.762$ (2) Å
$\beta = 103.76$ (2)°
$V = 1168.5$ (4) Å^3
$Z = 4$

$D_x = 1.127$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3850 reflections
$\theta = 1.0$–28.3°
$\mu = 0.24$ mm^{-1}
$T = 293$ (2) K
Prism, yellow
0.40 × 0.30 × 0.17 mm

Data collection

Nonius KappaCCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995, 1997)
$T_{\min} = 0.919$, $T_{\max} = 0.953$
6718 measured reflections

2843 independent reflections
1468 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.062$
$\theta_{\max} = 28.3°$
$h = -13 \rightarrow 8$
$k = -12 \rightarrow 13$
$l = -13 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.197$
$S = 1.06$
2843 reflections
122 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0847P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.44$ e Å^{-3}
$\Delta\rho_{\min} = -0.42$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.138 (8)

Table 1
Selected geometric parameters (Å, °).

S1—C3	1.697 (3)	N2—C3	1.351 (3)
N1—C3	1.342 (4)	N2—C2	1.391 (3)
N1—C1	1.388 (3)	C1—C2	1.342 (4)
C3—N1—C1	111.6 (2)	C1—C2—N2	108.8 (2)
C3—N2—C2	108.8 (2)	N1—C3—N2	105.9 (2)
C2—C1—N1	105.0 (2)		

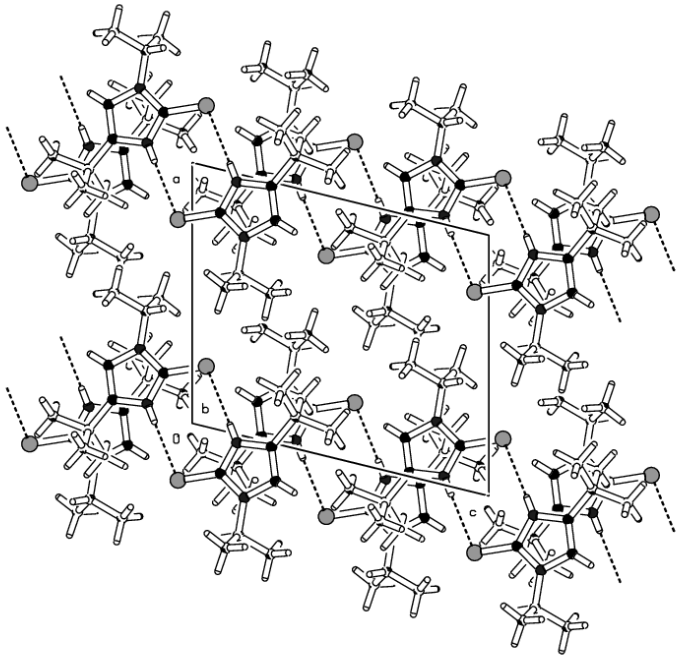

Figure 2
Crystal structure of the title compound, viewed along the *b* axis, showing the hydrogen-bonded dimers (dashed lines).

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1$^{\rm i}$	0.79	2.61	3.394 (3)	179

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

The positions of the methyl H atoms bound to atoms C9 and C10 were idealized (C—H = 0.96 Å), then refined with fixed isotropic displacement parameters [$U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$] as rigid groups allowed to rotate but not tip. All remaining H atoms were located in a difference Fourier map and refined using a riding model with fixed isotropic displacement parameters [$U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N,C})$]. The C—H distances are in the range 0.89–1.00 Å and the N—H distance os 0.79 Å.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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