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6-*tert*-Butyl-4-isopropylideneamino-3-methylsulfanyl-1,2,4-triazin-5(4*H*)-one

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Key indicators

Single-crystal X-ray study
T = 290 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.106
Data-to-parameter ratio = 15.4

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

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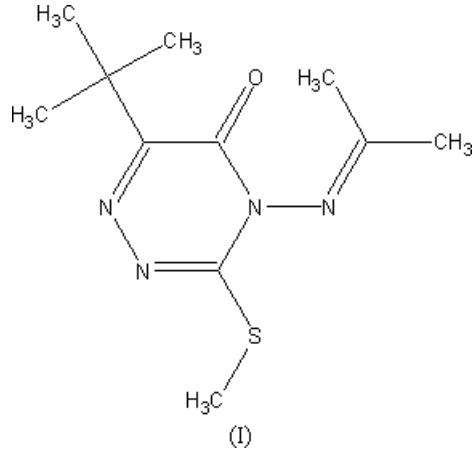
The title compound, $C_{11}H_{18}N_4OS$, a herbicide, crystallizes in an orthorhombic non-centrosymmetric space group. The crystal structure is stabilized by C—H···N and C—H···O intermolecular interactions.

Comment

An important aspect in the rational design of bioactive molecules involves relating chemical structure to biological activity (Lewis *et al.*, 1991). The conformation of the molecule is found to influence the levels of biological activity. Correlation of the results obtained from X-ray crystallography with biological activity has aided in the chemical design of a few active agrochemicals. The activity of a series of triazolyl ketone herbicides (Anderson *et al.*, 1983) has been investigated along with the fungicidal activities of *N*-phenylsuccinamides (Zenei *et al.*, 1988). In this paper, we report the structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. Relevant bond lengths, angles and torsion angles are given in Table 1. The crystal structure is stabilized by intermolecular C—H···O interactions [Etter symbol *C*(7); Bernstein *et al.*, 1995], forming molecular chains along the crystallographic *a* axis. Further C—H···N intermolecular interactions [Etter symbol *C*(7)] stabilize the packing of the molecules in the crystal structure, forming chains along the *c* axis, and thus sheets overall (Fig. 2).

Experimental

Metribuzin was obtained from Rallis India, Bangalore and a sample (0.05 g) was added to a beaker and was dissolved in a dichloromethane/acetone mixture (5 ml). Acetone was added to give the

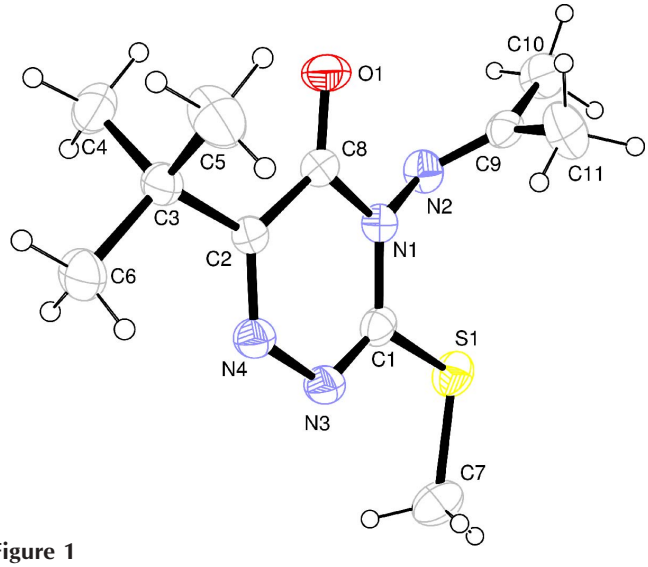


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 30% probability level.

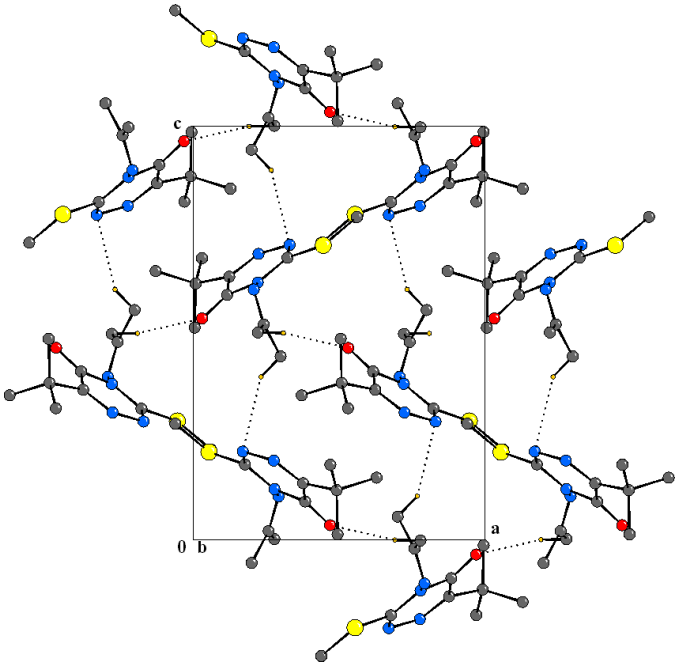


Figure 2
Packing diagram of (I), showing the C—H···N and C—H···O interactions (dashed lines) forming molecular sheets. H atoms have been omitted unless these are involved in hydrogen bonding.

corresponding Schiff base (yield 98%). Single crystals of (I) were obtained as blocks after complete evaporation of the solvent.

Crystal data

$C_{11}H_{18}N_4OS$
M_r = 254.36
Orthorhombic, $P2_12_12_1$
a = 9.797 (2) Å
b = 10.290 (3) Å
c = 13.856 (3) Å
V = 1396.9 (6) Å^3
Z = 4
D_x = 1.209 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 750 reflections
θ = 1.4–25.4°
μ = 0.22 mm^{-1}
T = 290 (2) K
Block, colorless
0.45 × 0.42 × 0.37 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.876, T_{max} = 0.922
10 073 measured reflections
2466 independent reflections
2196 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 25.0°
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.106
S = 1.15
2466 reflections
160 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 0.0414P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.012
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Absolute structure: Flack (1983), 1032 Friedel pairs
Flack parameter = 0.00 (10)

Table 1
Selected geometric parameters (Å, °).

C9—N2	1.273 (3)	N3—C1	1.293 (3)
N1—C1	1.369 (3)	N3—N4	1.385 (3)
N1—C8	1.390 (3)	N4—C2	1.294 (3)
N1—N2	1.426 (3)		
C8—N1—N2	118.0 (2)	C9—N2—N1	114.8 (2)
C7—S1—C1—N3	−3.2 (3)	N4—C2—C3—C4	−123.3 (3)
N4—C2—C3—C5	115.2 (3)	C8—C2—C3—C4	55.3 (3)
C8—C2—C3—C5	−66.2 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11B···N3^{i}	0.96	2.60	3.467 (4)	150
C10—H10B···O1^{ii}	0.96	2.35	3.289 (4)	166

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

All the methyl H atoms were constrained to an ideal geometry, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C), but were allowed to rotate freely about the C—C bond.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

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