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Diethyl (4-*tert*-butyl-1,3-thiazol-2-yl-aminomethylene)malonate

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Key indicators

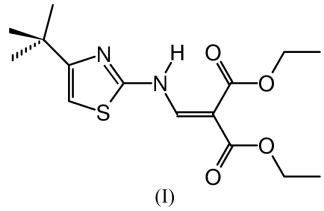
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.007 Å
H-atom completeness 96%
Disorder in main residue
R factor = 0.096
wR factor = 0.299
Data-to-parameter ratio = 17.3

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

The molecule of the title compound, $C_{15}H_{23}N_2O_4S$, is essentially planar, except for the *tert*-butyl group, with disorder of the aminomethylene atoms. These are equally disordered over two sites each, giving two alternative intramolecular hydrogen bonds from the partial amine H atoms to the adjacent carbonyl O atoms. An additional, intermolecular, C—H···O close contact is observed from the only thiazole ring H atom to one of the carbonyl O atoms.

Comment

2-Aminothiazole derivatives have a variety of pharmaceutical properties and recent experience has shown us that they are generally easy compounds to crystallize, or cocrystallize. There are currently 157 crystal structures (Cambridge Structural Database, September 2002 release; Allen, 2002) containing a 2-aminothiazole moiety. Only two of these structures have a 4-*tert*-butyl group, namely 4-*tert*-butyl-2-(*N*-methyl-*N*-phenylamino)thiazole-5-carbaldehyde (Gillon *et al.*, 1983) and *anti*-5-acetyl-2-dimethylamino-4-*tert*-butylthiazole (Caldwell *et al.*, 1987). The crystallographic quality of these two structures is very good, considering the potential for disorder in *tert*-butyl groups. Ironically, in the title compound, (I), the groups where disorder might be more likely, such as the *tert*-butyl and/or the ethyl esters, are relatively rigid, compared to the rest of the molecule. Large and very anisotropic displacement ellipsoids for three of the thiazole ring atoms (S1, C2 and N3) and the malonate atoms means that several atoms could be split into two positions each, but attempts to do so resulted in totally unrealistic bond distances and angles. The crystallographic results for (I) are of low precision, and a poor data set was the direct result of poor crystal quality. However, the resolvable disorder in this molecule and the probable unresolved disorder in the atoms with high displacement parameters is worth reporting.



(I)

The structure of (I) comprises an essentially planar molecule, except for the *tert*-butyl group, with resolved disorder of the aminomethylene atoms (Fig. 1). All the molecules in the unit cell lie essentially parallel to the *ab* plane. The aminomethylene atoms (N21 and C22) are both equally disordered

over two sites; thus, two alternative intramolecular hydrogen bonds exists from the partial amine H atoms to the adjacent carbonyl O atoms (Table 1). An additional C—H···O close contact is observed from the only thiazole ring H atom to one of the carbonyl O atoms in an adjacent molecule.

Experimental

The title compound was obtained from Key Organics Ltd and crystals were grown from iso-octane.

Crystal data

$C_{15}H_{23}N_2O_4S$
M_r = 327.41
Monoclinic, $C2/c$
a = 19.663 (4) Å
b = 13.177 (3) Å
c = 13.708 (3) Å
β = 103.69 (3)°
V = 3450.8 (13) Å^3
Z = 8
D_x = 1.257 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7703 reflections
θ = 2.9–27.5°
μ = 0.21 mm^{-1}
T = 150 (2) K
Needle, colourless
0.34 × 0.10 × 0.08 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.933, T_{max} = 0.984
12347 measured reflections
3916 independent reflections
1830 reflections with $I > 2\sigma(I)$
R_{int} = 0.113
θ_{max} = 27.5°
$h = -25 \rightarrow 25$
$k = -17 \rightarrow 17$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.096
$wR(F^2)$ = 0.300
S = 1.16
3916 reflections
226 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1206P)^2 + 1.7968P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.94 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0067 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N21A—H21A···O28	0.88	2.26	2.931 (10)	133
N21B—H21B···O24	0.88	2.33	2.913 (9)	123
C5—H5···O28^{i}	0.95	2.44	3.363 (7)	163

Symmetry code: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$.

All H atoms were included in the refinement, at calculated positions, as riding models, with X—H set to 0.88 (N—H), 0.95 (aromatic), 0.99 (CH_2) or 0.98 Å (CH_3), and U_{iso}(H) = 1.25U_{eq}(X). The high R_{int} value is the result of weak high-angle data.

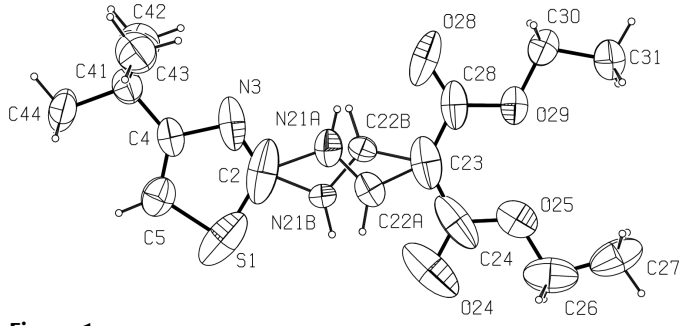


Figure 1
The molecular structure and atom-numbering scheme for the title compound, showing 50% probability ellipsoids and the resolved twofold disorder.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON*97 (Spek, 1997); software used to prepare material for publication: *SHELXL*97.

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