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Ethyl 2-(2-chloro-1,4-dihydro-1,4-dioxonaphthalen-3-ylamino)-4-phenylthiazole-5-carboxylate

Daniel E. Lynch[a]* and Ian McClenaghan[b]

[a]School of Science and the Environment, Coventry University, Coventry CV1 5FB, England, and [b]Key Organics Ltd, Highfield Industrial Estate, Camelford, Cornwall PL32 9QZ, England

Correspondence e-mail: apx106@coventry.ac.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.094
wR factor = 0.251
Data-to-parameter ratio = 13.3

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

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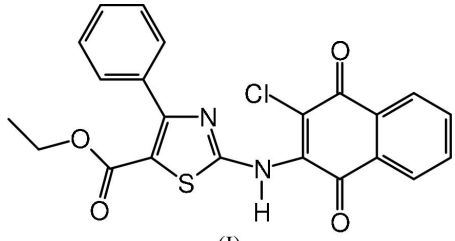
The structure of the title compound, $C_{22}H_{15}ClN_2O_4S$, comprises non-planar molecules that form a one-dimensional hydrogen-bonded chain *via* a single N—H···O interaction, which runs parallel to the *b* axis. The dihedral angle between the thiazole and quinone rings is 50.43 (7)° and the dihedral angle between the thiazole and the phenyl rings is 52.4 (1)°.

Comment

The title compound, (I), was prepared with the intention of merging two separate studies that we have recently undertaken. One study involved the synthesis and structural properties of 2-substituted 3-chloro-1,4-naphthoquinones (Lynch & McClenaghan, 2002; 2003), while the other involved 2-aminothiazoles. From the latter study came the structure of the thiazole derivative used to prepare (I), *viz*. ethyl 2-amino-4-phenylthiazole-5-carboxylate (Lynch & McClenaghan, 2000). By bringing together the two series of molecules, we are interested in examining the combined structural aspects of the resultant covalently linked products, especially considering the forced proximity of one N—H hydrogen-bond donor with five hydrogen-bond acceptors (*viz*. two O atoms, one N atom, one Cl atom and one S atom). The structure of (I) comprises non-planar molecules, the dihedral angle between the thiazole and quinone rings being 50.43 (7)° and the dihedral angle between the thiazole and phenyl rings being 52.4 (1)°. The equivalent dihedral angle in the parent thiazole molecule is 42.41 (6)°.



(I)

Molecules of (I) form a one-dimensional hydrogen-bonded chain *via* a single N—H···O interaction [graph set *C*(6); Etter, 1990], which runs parallel to the *b* axis; hydrogen-bonding geometry is given in Table 1. A close contact C25—H25···O21^{i} [C···O^{i} = 3.165 (3) Å, H···O^{i} = 2.22 Å and C—H···O^{i} = 172°; symmetry code: (i) *x*, 1 + *y*, *z*] exists adjacent to the N—H···O interaction and thus completes an $R_2^2(10)$ graph-set motif.

Experimental

The title compound was obtained from Key Organics Ltd and crystals were grown from an ethanol solution.

Crystal data

$C_{22}H_{15}ClN_2O_4S$
M_r = 438.87
Monoclinic, $P2_1/c$
a = 19.191 (5) Å
b = 7.719 (2) Å
c = 12.640 (3) Å
β = 94.845 (18)°
V = 1865.6 (8) Å^3
Z = 4
D_x = 1.563 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4585 reflections
θ = 2.9–27.5°
μ = 0.35 mm^{-1}
T = 120 (2) K
Plate, orange
0.18 × 0.14 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.939, T_{max} = 0.993
34099 measured reflections
3678 independent reflections
3672 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 26.0°
$h = -23 \rightarrow 23$
$k = -9 \rightarrow 9$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.094
$wR(F^2)$ = 0.251
S = 1.14
3678 reflections
276 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0933P)^2 + 12.2716P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N21—H21···O24^{i}	0.86 (2)	2.27 (2)	3.066 (3)	154 (2)

Symmetry code: (i) $x, y-1, z$.

The amino H atom was located in a difference Fourier synthesis and its positional parameters were refined. Other H atoms were included in the refinement at calculated positions in the riding-model approximation, with C—H distances of 0.95 (aromatic H atoms), 0.98 (CH_3 H atoms) and 0.99 Å (CH_2 H atoms). The isotropic displacement parameters for all H atoms were set equal to 1.25U_{eq} of the carrier atom. The high R value in this structure was a direct consequence of poor data from poor-quality twinned crystals; the non-merohedral twinning was refined as two components with ratio 0.5207 (8):0.4793 (8).

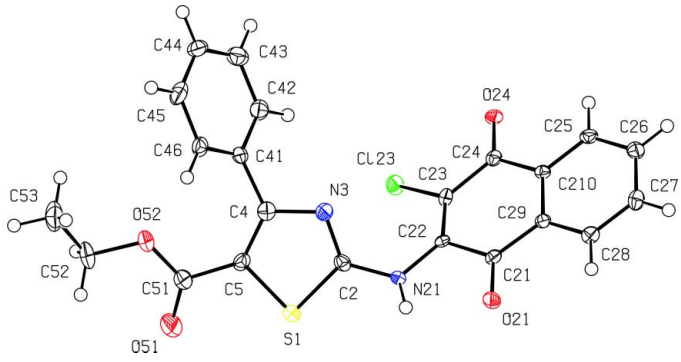


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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