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Ethyl 3-{[(2-chlorophenyl)sulfonyl]imino}-2-{[(3,5-dimethoxyphenyl)amino]carbonyl}-3-(methylsulfanyl)propanoate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.010 Å
Disorder in main residue
R factor = 0.043
wR factor = 0.152
Data-to-parameter ratio = 14.9

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

The title compound, $C_{21}H_{23}ClN_2O_7S_2$, is a representative of moderately active hydroxyacid synthase inhibitors. The structure has a sulfonylimine bond [—N═C(—SMe)—CH] and not sulfonylamino [—NH—C(—SMe)═C], contrary to expectations. There is a strong intramolecular hydrogen bond between the imine H and the acid carbonyl group, and a weaker one between the imine carbonyl group and a benzene H atom.

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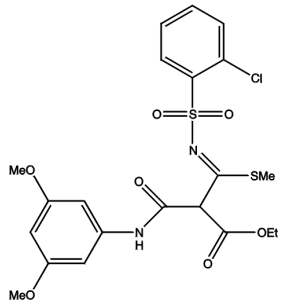
Comment

This report is part of a general study on acetohydroxyacid synthase inhibitors (McFadden *et al.*, 1993). The title compound, (I), was reported in that paper as a molecule with a double bond between atoms C2 and C3. However, a crystal structure determination (Fig. 1 and Table 1) shows that the double bond occurs between atoms N1 and C2 [1.339 (6) Å], indicating a minimum energy structure with a sulfonylimine link. There are intramolecular N1—H1···O32 and C12—H12···O21 hydrogen bonds in (I). Compared with the inactive compound 3-[(2-chlorophenyl)sulfonylamino]-2-cyano-*N*-(3,5-dimethoxyphenyl)-3-methylsulfanyl-2-propenamide (Kennard *et al.*, 2003), compound (I) has different torsion angles [N1—C2—C3—C4 = 155.5 (5) and −179.67 (17)°, C2—C3—C4—N5 = −69.2 (7) and −3.9 (3)°, and C3—C4—N5—S6 = 0.7 (9) and 170.64 (15)°].



(I)

Experimental

The synthesis of (I) has been reported by McFadden *et al.* (1993).

Crystal data

$C_{21}H_{23}ClN_2O_7S_2$
M_r = 514.98
Monoclinic, $P2_1/n$
a = 8.546 (4) Å
b = 24.166 (3) Å
c = 12.057 (8) Å
β = 104.67 (2)°
V = 2409 (2) Å^3
Z = 4
D_x = 1.420 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–12°
μ = 0.38 mm^{-1}
T = 293 (2) K
Plate, colourless
0.30 × 0.05 × 0.05 mm

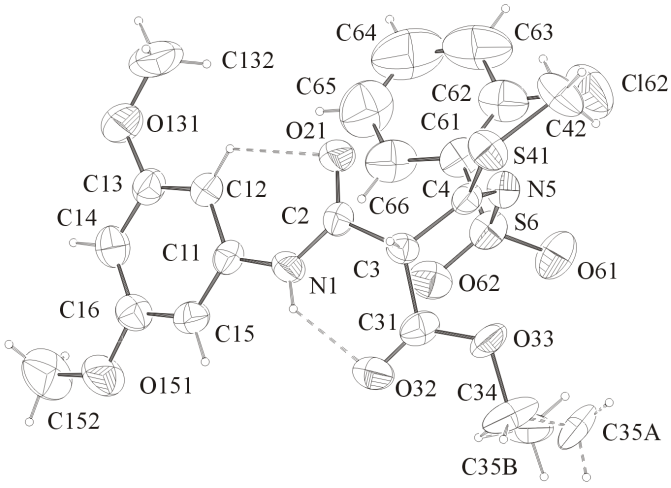


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level. The occupation factors of atoms C35*A* and C35*B* are 40 and 60%, respectively.

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.933, T_{max} = 0.979
4570 measured reflections
4233 independent reflections
1419 reflections with $I > 2\sigma(I)$

R_{int} = 0.037
θ_{max} = 25.0°
$h = -4 \rightarrow 10$
$k = -11 \rightarrow 28$
$l = -14 \rightarrow 13$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.152
S = 0.91
4233 reflections
307 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0626P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.011
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—C3	1.546 (6)	C3—C4	1.540 (7)
C12—C11—N1—C2	−11.3 (8)	C3—C4—N5—S6	1.0 (8)
C11—N1—C2—C3	−172.8 (5)	C4—N5—S6—C61	112.7 (5)
N1—C2—C3—C4	155.6 (5)	N5—S6—C61—C62	65.8 (5)
C2—C3—C4—N5	−69.4 (7)		

The terminal atom C35 of the ethyl group is disordered over two sites, C35*A* and C35*B*. Their occupation factors were assigned, from refinement, to be 40 and 60%, respectively. The ratios of atomic displacement parameters for the various directions for C35*A* are large and indicate serious disorder.

Data collection: *SDP* (Frenz, 1985); cell refinement: *SDP*; data reduction: *WinGX* (Farrugia, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97; molecular graphics: *PLATON*98 (Spek, 1988); software used to prepare material for publication: *SHELXL*97.

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