Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*Z*)-2-[4-(2-Chlorophenyl)-1,3-dithiolan-2-ylidene]-2-(imidazol-1-yl)acetonitrile

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.124
Data-to-parameter ratio = 15.5

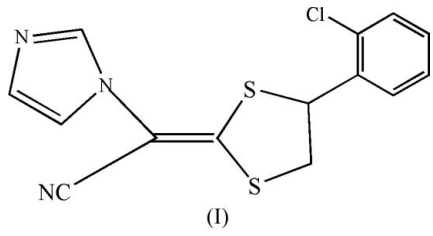
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 24 June 2006
Accepted 27 June 2006

In the molecule of the title compound, $C_{14}H_{10}ClN_3S_2$, the dithiolane ring is not planar. Intermolecular C—H···N hydrogen bonds linking the molecules may be effective in stabilizing the crystal structure.

Comment

The title compound, (I), has potent activity against a wide variety of pathogenic fungi, such as dermatophytes, candidiasis and trichophyton. We report here the crystal structure of (I).



(I)

In the molecule of the title compound, (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

Ring B (S1/S2/C6–C8) is not planar, with deviations of 0.1460 (3), −0.1479 (4), −0.0037 (3), 0.3166 (3) and −0.3109 (4) Å for atoms S1, S2, C6, C7 and C8, respectively, from the least-squares plane. Rings A (N1/N2/C1–C3) and C (C9–C14) are, of course, planar and the dihedral angle between them is 75.31 (4)°.

As can be seen from the packing diagram (Fig. 2), intermolecular C—H···N hydrogen bonds [H1*B*···N3^{i} = 2.50 Å, C1···N3^{i} = 3.423 (4) Å and C1—H1*B*···N3^{i} = 173°, and H2*A*···N3^{ii} = 2.57 Å, C2···N3^{ii} = 3.456 (4) Å and C2—H2*A*···N3^{i} = 160°; symmetry codes: (i) $x-1, y, z$; (ii) $x-1, \frac{1}{2}-y, \frac{1}{2}+z$] link the molecules and may be effective in the stabilization of the crystal structure. Dipole–dipole and van der Waals interactions are also effective in the molecular packing.

Experimental

DMF (120 ml) was placed in a three-necked round-bottomed flask fitted with a mechanical stirrer, dropping funnel and reflux condenser. The system was put in an ice bath and stirred, then sodium hydride (8.0 g, 0.2 mol) was added. Imidazole (13.6 g, 0.2 mol) was dissolved in DMF (40 ml), and the mixture was added dropwise slowly, and stirred at room temperature for 30 min. Chloroacetonitrile (15.1 g, 0.2 mol) was added and stirred for 4 h and cooled in an ice bath. Potassium hydroxide (22.4 g, 0.2 mol) and carbon disulfide (15.2 g,

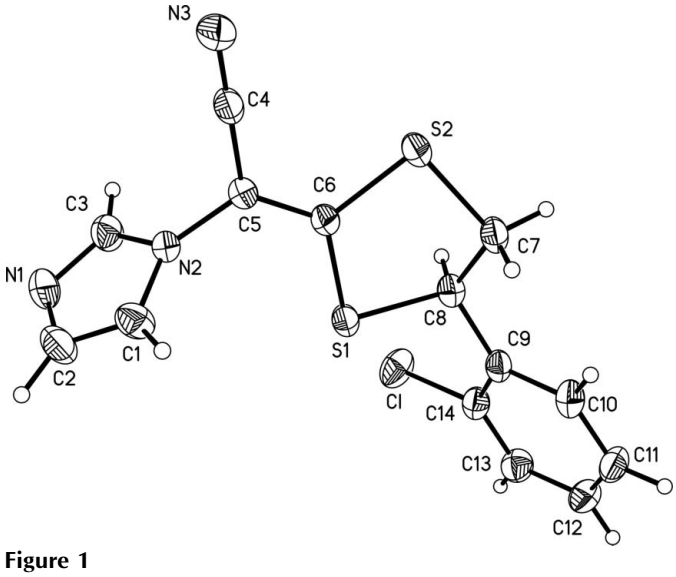


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

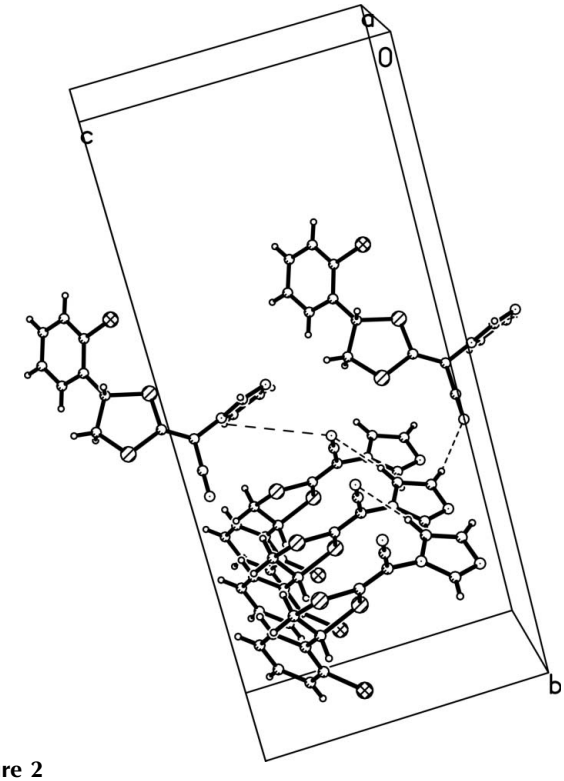


Figure 2
A patial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

0.2 mol) were added in turn, and stirred for 30 min, and then stirred at room temperature for an additional 4 h and cooled in an ice bath. 2-(1,2-Dibromoethyl)-1-chlorobenzene (60.0 g, 0.2 mol) was added dropwise and stirred for 3 h at room temperature. The red–brown reaction solution was poured into ice–water (400 ml). Dichloromethane (200 ml) was used to extract the mixture, the organic layer was combined, then washed successively with saturated sodium carbonate solution (200 ml) and water (200 ml), dried by anhydrous magnesium sulfate, filtered and distilled under reduced pressure until dry. Ethyl acetate (250 ml) and dry ethanol (50 ml) were added to the reddish brown oily residue and heated until the mixture had dissolved. Activated carbon (1.0 g) was added, and the solution was refluxed for 15 min and filtered hot. When the filtrate had cooled, it was put into an ice-box for 24 h. The mixture was filtered and the solid was dried, giving 32.0 g of the yellow crude product. Recrystallization from a mixture of ethyl acetate and petroleum ether (10:1) gave the title compound as yellow crystals (yield 26.0 g, 43.3%, m.p. 413–415 K). It was recrystallized from dry ethanol by slow evaporation over a period of 15 d.

Crystal data

$C_{14}H_{10}ClN_3S_2$
M_r = 319.82
Monoclinic, $P2_1/c$
a = 5.7930 (12) Å
b = 23.652 (5) Å
c = 10.466 (2) Å
β = 91.66 (3)°
V = 1433.4 (5) Å^3
Z = 4
D_x = 1.482 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.55 mm^{-1}
T = 294 (2) K
Block, yellow
0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.810, T_{max} = 0.898
3081 measured reflections
2803 independent reflections
2034 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 26.0°
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.124
S = 1.03
2803 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.P)^2 + 0.6P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.094 (12)

H atoms were positioned geometrically, with C—H = 0.93, 0.98 and 0.97 Å for aromatic, methine and methylene H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

The authors thank the Center of Testing and Analysis, Nanjing University, for support.

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