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2-[3-Acetyl-5-(3,6-dichloropyridin-2-yl)-2,3-dihydro-1,3,4-oxadiazol-2-yl]phenyl acetate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.116
Data-to-parameter ratio = 16.7

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

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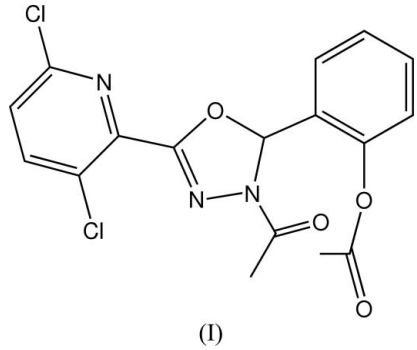
The title compound, $C_{17}H_{13}Cl_2N_3O_4$, shows the substituted pyridine ring to be coplanar with the oxadiazoline ring but the third ring to be normal to this. Chains mediated by C—H···O interactions are linked to two other chains *via* C—H···Cl interactions and to another two chains *via* C—H···π interactions.

Comment

Oxadiazolines represent a class of heterocycle that, although known for over a century, is rather limited in number. These are partially reduced forms of well known oxadiazoles. Recently, oxadiazolines have received increased attention owing to their usefulness as synthetic intermediates (Charmier *et al.*, 2004; Moustafa, 2003) and promising biological activity. For example, oxadiazolines are reported to possess anti-tumour (Chimirri *et al.*, 1996), anti-HIV (Chimirri *et al.*, 1994), antifungal (Singh & Hasan, 2002), anti-inflammatory (Tinperciuc *et al.*, 1999) and anticonvulsant (Dogan *et al.*, 1998) properties. 3-Acetyl-2,5-disubstituted-1,3,4-oxadiazolines are also known to exhibit antimicrobial activity (Hassan *et al.*, 1983; Khalil *et al.*, 1993). It was in this context that the title compound, (I), was prepared.



(I)

The molecule of (I) (Fig. 1) is twisted about the C2—C10 bond, as seen in the O1/C2/C10/C11 torsion angle of 58.02 (19)°. This twist most likely arises to relieve the putative strain between the N3-acyl and C11-acetyl groups. The consequence of this twist is that the C10–C15 ring is almost normal to the oxadiazoline ring, as seen in the dihedral angle between their least-squares planes of 89.11 (8)°. By contrast, the oxadiazoline is effectively coplanar with the N1/C3–C7 ring [dihedral angle = 4.58 (8)°]. Selected interatomic parameters are collected in Table 1 and these show that the N2—C1 bond distance corresponds to a double bond, indicating limited delocalization of π-electron density over the oxadiazoline ring despite its planarity; maximum deviation of

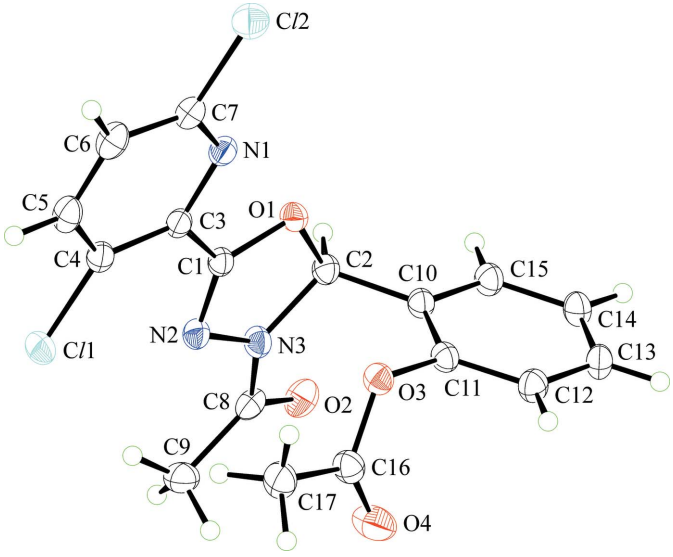


Figure 1
The atom-labelling scheme for (I), showing 50% probability displacement ellipsoids.

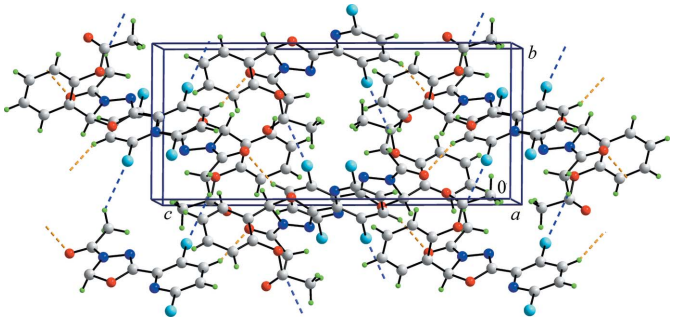


Figure 2
Chain formation in (I), viewed approximately down the *a* axis (Crystal Impact, 2006). Colour code: Cl (cyan), O (red), N (blue), C (grey) & H (green). Gold and blue dashed lines indicate C—H···O and C—H···Cl hydrogen-bonding interactions, respectively.

0.0185 (16) Å for atom C2. The acyl group is coplanar with the oxadiazoline ring, as seen in the N2—N3—C8—O2 torsion angle of 171.00 (14)°, but the acetyl group is normal to the C10–C15 ring, as evidenced by the C10—C11—O3—C16 torsion angle of −109.07 (17)°.

The crystal structure is stabilized by C—H···O, C—H···Cl and C—H···π interactions. A chain along the *c*-axis direction is mediated by C—H···O interactions, highlighted as orange dashed lines in Fig. 2, so that C5—H5···O2^{i} is 2.29 Å, C5···O2^{i} is 3.182 (2) Å and the angle at H is 155° [symmetry code: (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$]. These are linked to centrosymmetrically related chains *via* C—H···Cl interactions aligned along the *b*-axis direction and highlighted as blue dashed lines in Fig. 2, with C9—H9*c*···Cl1^{ii} of 2.79 Å, C9···Cl1^{ii} of 3.620 (2) Å and angle at H of 143° [symmetry code: (ii) $1 - x, 1 - y, 1 - z$]. Atom N2 does not form an intermolecular interaction but forms an intramolecular C9—H9*b*···N2 contact of 2.41 Å, so that C9···N2 is 2.836 (3) Å and the angle at H is 106°. Finally, C—H···π interactions are evident, indicated by '*a*' in Fig. 3, that link a further two chains to the original so that each chain is surrounded by four chains in total. Here, the C12—H···*Cg*^{iii} distance (*Cg* is the centroid of ring C10–C15) is 2.79 Å, with an angle of 172° at H [symmetry code: (iii) $-x, \frac{1}{2} + y, \frac{1}{2} - z$].

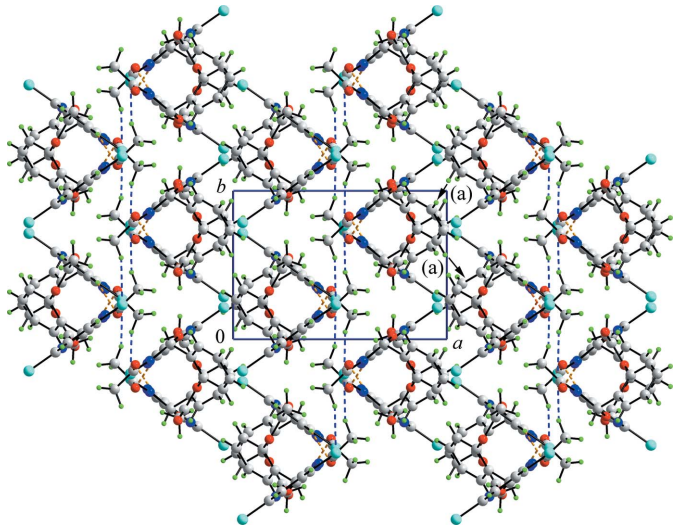


Figure 3
The crystal packing in (I), viewed down the *c* axis. Colour code and hydrogen bonds as for Fig. 2.

Experimental

Compound (I) was prepared in accord with the literature procedure (Yale *et al.*, 1953). Colourless crystals were obtained by slow evaporation of an ethyl acetate solution of (I) after 4 d at room temperature (m.p. 446 K).

Crystal data

$C_{17}H_{13}Cl_2N_3O_4$
M_r = 394.20
Monoclinic, $P2_1/c$
a = 11.839 (4) Å
b = 7.917 (3) Å
c = 19.255 (7) Å
β = 105.658 (6)°
V = 1737.6 (10) Å^3
Z = 4
D_x = 1.507 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 173 (2) K
Plate, colourless
0.35 × 0.35 × 0.10 mm

Data collection

Rigaku AFC12K/SATURN724 diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.851, T_{max} = 1
45244 measured reflections
3956 independent reflections
3911 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.116
S = 1.16
3956 reflections
237 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0598P)^2 + 0.6091P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C4	1.7245 (17)	O4—C16	1.186 (2)
Cl2—C7	1.7423 (18)	N1—C7	1.311 (2)
O1—C1	1.3675 (17)	N1—C3	1.348 (2)
O1—C2	1.4414 (17)	N2—C1	1.282 (2)
O2—C8	1.218 (2)	N2—N3	1.3871 (17)
O3—C16	1.373 (2)	N3—C8	1.363 (2)
O3—C11	1.4037 (18)	N3—C2	1.4727 (19)
C1—O1—C2	106.63 (11)	N2—N3—C8	124.06 (13)
C1—N2—N3	104.40 (12)	C2—N3—C8	124.02 (13)
N2—N3—C2	111.64 (12)		

The C-bound H atoms were included in the riding-model approximation with C—H = 0.95–0.98 Å, and U_{iso}(H) = 1.5U_{eq}(me-eq(methyl C) or 1.2U_{eq}(C).

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Crystal Impact, 2006); software used to prepare material for publication: *SHELXL97*.

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