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4-(2,2-Dichloro-*N*-methylacetamido)phenyl furan-2-carboxylate

S. Naveen,[a] Dinesh Manvar,[b] Anamik Shah,[b] M. A. Sridhar[a]* and J. Shashidhara Prasad[a]

[a]Department of Studies in Physics, Mansagangotri, University of Mysore, Mysore 570 006, India, and [b]Department of Chemistry, Saurashtra University, Rajkot 360 005, India

Correspondence e-mail: mas@physics.uni-mysore.ac.in

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.143
Data-to-parameter ratio = 13.1

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

In the title compound, $C_{14}H_{11}Cl_2NO_4$, the dihedral angle between the two aromatic rings is 47.39 (13)°. The crystal packing is stabilized by C—H···O hydrogen bonds.

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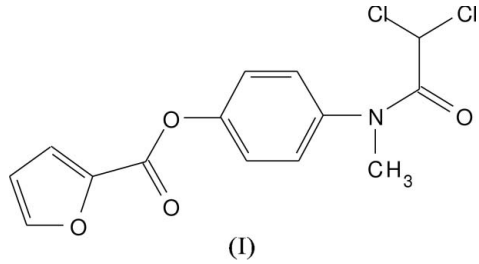
Comment

Amoebiosis is a major public health problem in tropical and subtropical countries (McAuley *et al.*, 1992). Diloxanide furoate, (I), is the mainstay for treating asymptomatic cyst carriers and one of the oldest dichloroacetamide derivative drugs for the treatment of amoebiosis. A crystal structure determination was carried out in order to elucidate the molecular conformation.



(I)

A perspective view of (I) is shown in Fig. 1. In (I), the furan ring is planar. The dihedral angle between the furan ring and the benzene ring is 47.39 (13)°. The keto group is almost coplanar with the furan ring, as indicated by the C1—C2—C6—O7 torsion angle of −8.5 (4)°, and is in a synperiplanar conformation [O7—C6—O8—C9 = 8.3 (3)°]. The other keto group adjacent to the *N*-methyl unit is in an antiperiplanar conformation, as indicated by the C12—N15—C17—O18 torsion angle of 177.9 (2)°. The 2,2-dichloroacetylmethylamino group at the 4 position of the benzene ring is rotated 80.3 (1)° from the plane of the benzene ring. The crystal packing is stabilized by C—H···O hydrogen bonding (Table 1). The molecules form hydrogen-bonded dimers.

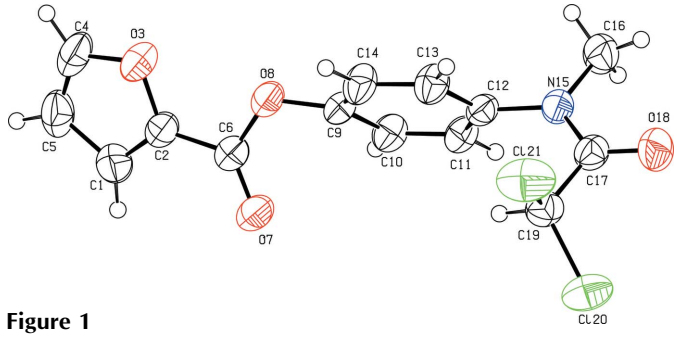


Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids.

Experimental

Diloxanide furoate (1.5 g) was dissolved in methanol (30 ml). Charcoal (3.5 g) was added and the mixture was stirred with heating for 6 minutes on a Deepali Stirrer (MS-4) at 1150 r.p.m. The hot solution was filtered through Whatmann 42 filter paper and kept in a slightly open, stoppered conical flask. Rectangular pale-green crystals grew by thin-film evaporation.

Crystal data

$C_{14}H_{11}Cl_2NO_4$
M_r = 328.14
Monoclinic, $P2_1/n$
a = 8.334 (5) Å
b = 10.778 (5) Å
c = 16.412 (10) Å
β = 103.761 (2)°
V = 1431.9 (14) Å^3
Z = 4
D_x = 1.522 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 295 (2) K
Block, pale green
0.25 × 0.24 × 0.22 mm

Data collection

MacScience DIPLabo 32001 diffractometer
ω scans
Absorption correction: none
4433 measured reflections
2524 independent reflections
2209 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.143
S = 1.20
2524 reflections
192 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0816P)^2 + 0.2846P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.056 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O18^{i}	0.93	2.41	3.285 (4)	156

Symmetry code: (i) $x-\frac{3}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

H atoms were placed at idealized positions and allowed to ride on their parent atoms, with C—H = 0.92—0.98 Å and N—H = 0.86 Å; U_{iso}(H) values were set equal to xU_{eq}(carrier atom), where x = 1.5 for methyl H atoms and x = 1.2 for all other H atoms.

Data collection: *XPRESS* (MacScience, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *PLATON* (Spek, 2003).

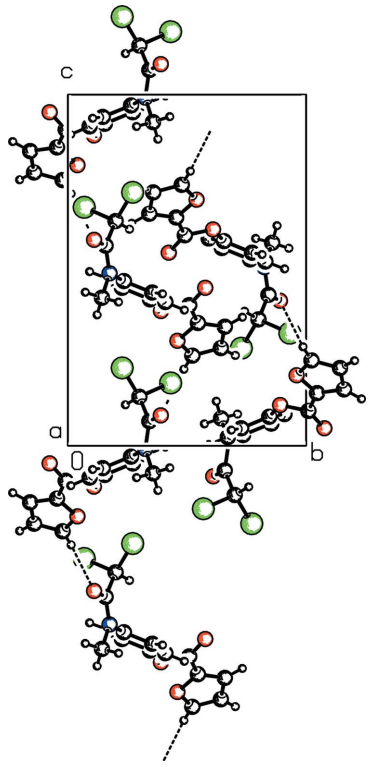


Figure 2
The packing of (I), viewed down the c axis. Dashed lines indicate hydrogen bonds.

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