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1,5-Bis(4-chlorophenyl)-3-(2-chloroquinolin-3-yl)pentane-1,5-dione: sheets of $R_4^4(26)$ rings built from C—H···N and C—H···O hydrogen bonds

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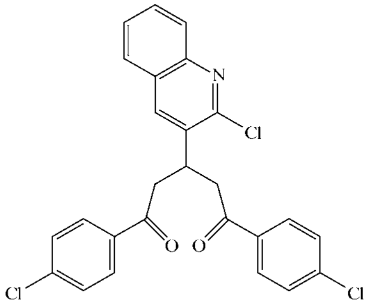
Molecules of the title compound, $C_{26}H_{18}Cl_3NO_2$, are linked into sheets of $R_4^4(26)$ rings by a combination of C—H···N and C—H···O hydrogen bonds.

Comment

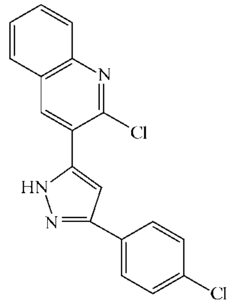
With the aim of developing new classes of fused heterocyclic systems, we have prepared a range of novel chalcones appropriately functionalized for use as intermediates. The reactions used to prepare such chalcones involve methyl aryl ketones and aryl or heteroaryl aldehydes. We report here the structure of the title compound, (I) (Fig. 1), obtained in low yield as a by-product in the preparation of the pyrazolylquinoline (II) *via* the corresponding chalcone (III), formed by the reaction between 2-chloroquinoline-3-carbaldehyde and 4-chlorophenyl methyl ketone.

The bond distances within the quinoline portion of the molecule (Table 1) show evidence for significant bond fixation; the N31—C32 bond is thus significantly shorter than N31—C38*A*, while the C33—C34, C35—C36 and C37—C38 bonds are all significantly shorter than the other peripheral C—C bonds. The two independent 4-chlorobenzoylmethylene components adopt different conformations relative to the quinoline component so that the molecules have no internal symmetry and hence are chiral; however, the centrosymmetric space group *Pbca* accommodates equal numbers of the two enantiomorphic forms.

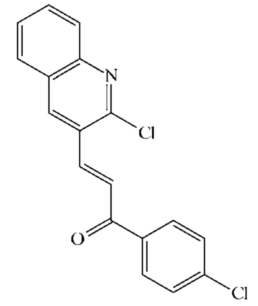
The molecules are linked into sheets by two hydrogen bonds, one each of the C—H···N and C—H···O types (Table 2), and the sheet formation is readily analysed in terms of two one-dimensional substructures. Quinolinyl atom C34 in the molecule at (x, y, z) acts as a hydrogen-bond donor to atom N31 in the molecule at ($-\frac{1}{2} + x$, y, $\frac{1}{2} - z$), so forming a $C(5)$ (Bernstein *et al.*, 1995) chain running parallel to the [100] direction and generated by the a-glide plane at $z = \frac{1}{4}$ (Fig. 2). In addition, quinolinyl atom C37 in the molecule at (x, y, z) acts as a hydrogen-bond donor to atom O17 in the molecule at ($1 - x$, $-\frac{1}{2} + y$, $\frac{1}{2} - z$), so forming a $C(11)$ chain running parallel to the [010] direction and generated by the 2_1 screw axis along ($\frac{1}{2}$, y, $\frac{1}{4}$) (Fig. 3).



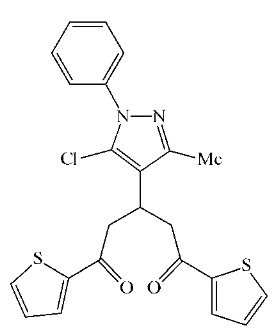
(I)



(II)



(III)



(IV)

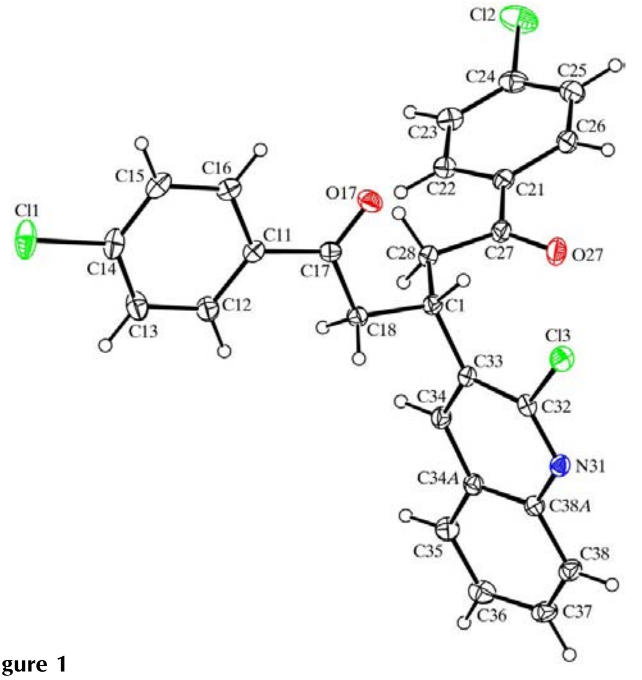


Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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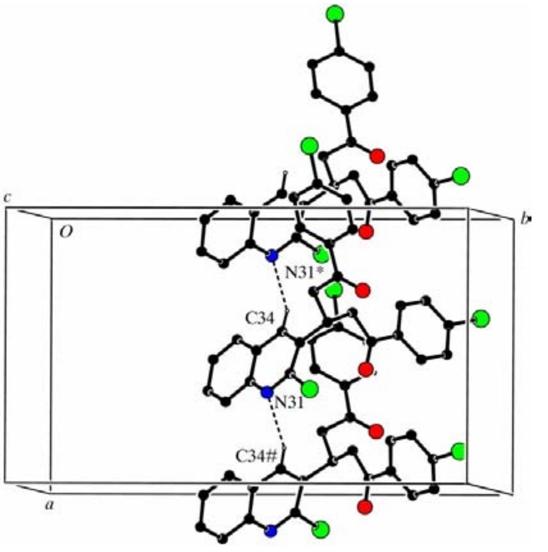

Figure 2
Part of the crystal structure of (I), showing the formation of a hydrogen-bonded $C(5)$ chain along [100]. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(-\frac{1}{2} + x, y, \frac{1}{2} - z)$ and $(\frac{1}{2} + x, y, \frac{1}{2} - z)$, respectively.

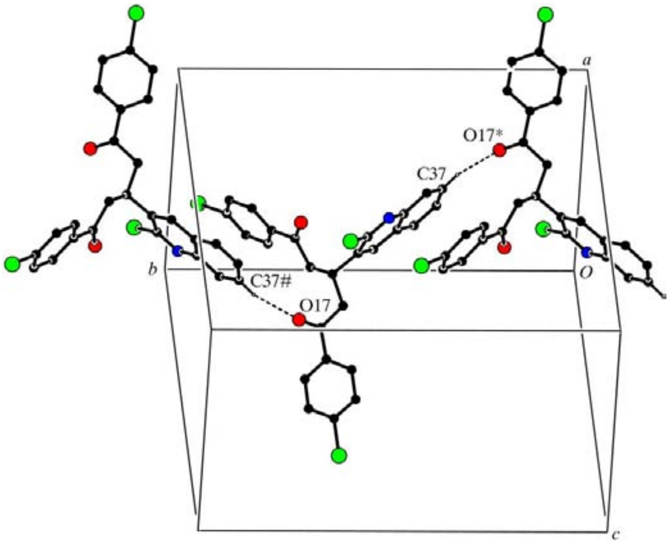

Figure 3
Part of the crystal structure of (I), showing the formation of a hydrogen-bonded $C(11)$ chain along [010]. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(1 - x, -\frac{1}{2} + y, \frac{1}{2} - z)$ and $(1 - x, \frac{1}{2} + y, \frac{1}{2} - z)$, respectively.

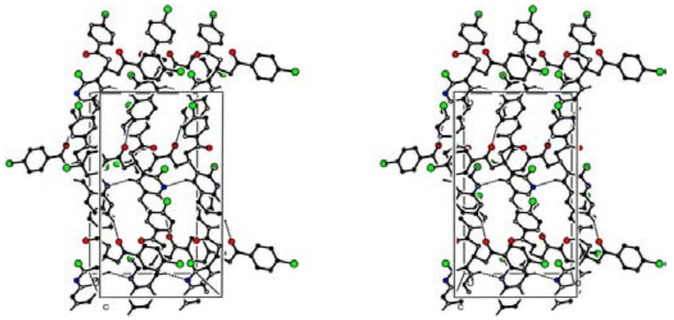

Figure 4
A stereoview of part of the crystal structure of (I), showing the formation of a (001) sheet built from $R_4^4(26)$ rings. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

The combination of the [100] and [010] chains generates a (001) sheet in the form of a (4,4)-net (Batten & Robson, 1998) built from a single type of $R_4^4(26)$ ring, lying in the domain $-0.02 < z < 0.52$, and generated by the glide plane and screw axes at $z = \frac{1}{4}$ (Fig. 4). A second sheet, related to the first by inversion, lies in the domain $0.48 < z < 1.02$, and is generated by the glide plane and screw axes at $z = \frac{3}{4}$.

The only direction-specific interaction between adjacent sheets is a rather long C—H$\cdots$O contact with aryl atom C15 as the donor (Table 2) but whose H$\cdots$O distance is close to the van der Waals limit; this interaction is therefore probably of little or no structural significance.

The molecular constitution of (I) has some resemblance to that of the thienyl compound (IV), but the supramolecular arrangement is entirely different in (IV), where the molecules are linked into cyclic centrosymmetric dimers by paired C—H$\cdots\pi$(thienyl) hydrogen bonds (Trilleras *et al.*, 2005).

Experimental

Hydrazine hydrate (0.70 g of a 55% aqueous solution, 12 mmol) was added dropwise to a solution of (*E*)-1-(4-chlorophenyl)-3-(2-chloroquinolin-3-yl)prop-2-en-1-one, (III) (2.3 g, 7 mmol), in methanol (40 ml), and the resulting mixture was then stirred at room temperature for 15 min. The solid product was collected by filtration and washed with cold methanol to give 2-chloro-3-[3-(4-chlorophenyl)-1*H*-pyrazol-5-yl]quinoline, (II) (2.2 g, 88% yield). Evaporation of the filtrate yielded crystals of (I) suitable for single-crystal X-ray diffraction (yield 10%, m.p. 505–506 K). MS (EI 70 eV), m/z (%) 481 (5, M^+), 328 (64, $[M - \mathrm{ClC_6H_4COCH_2}]^+$), 292 (62), 139 (100).

Crystal data

$C_{26}H_{18}Cl_3NO_2$
$M_r = 482.76$
Orthorhombic, *Pbca*
$a = 11.2405$ (2) Å
$b = 18.8738$ (3) Å
$c = 21.0529$ (3) Å
$V = 4466.39$ (12) Å^3
$Z = 8$
$D_x = 1.436$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 5112 reflections
$\theta = 3.4$–$27.5°$
$\mu = 0.44$ mm^{-1}
$T = 120$ (2) K
Block, colourless
0.20 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.922$, $T_{\max} = 0.938$
52455 measured reflections
5112 independent reflections

3921 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.048$
$\theta_{\max} = 27.5°$
$h = -14 \rightarrow 14$
$k = -21 \rightarrow 24$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.116$
$S = 1.05$
5112 reflections
289 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 2.4672P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.41$ e Å^{-3}
$\Delta\rho_{\min} = -0.58$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N31—C32	1.302 (2)	C35—C36	1.369 (3)
N31—C38*A*	1.368 (2)	C36—C37	1.402 (3)
C32—C33	1.422 (2)	C37—C38	1.369 (3)
C33—C34	1.370 (3)	C38—C38*A*	1.416 (3)
C34—C34*A*	1.414 (3)	C34*A*—C38*A*	1.409 (3)
C34*A*—C35	1.418 (3)	C32—Cl3	1.7463 (18)
C32—C33—C1—C18	−110.03 (19)	C32—C33—C1—C28	126.64 (18)
C33—C1—C18—C17	160.82 (15)	C33—C1—C28—C27	−67.55 (19)
C1—C18—C17—C11	171.11 (15)	C1—C28—C27—C21	−176.29 (15)
C18—C17—C11—C12	3.3 (3)	C28—C27—C21—C22	−16.4 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15···O17^{i}	0.95	2.59	3.502 (2)	160
C34—H34···N31^{ii}	0.95	2.39	3.244 (2)	150
C37—H37···O17^{iii}	0.95	2.45	3.330 (2)	155

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

The space group *Pbca* was uniquely assigned from the systematic absences. All H atoms were located from difference maps and then treated as riding atoms, with C—H distances of 0.95 (aromatic), 0.99 (CH_2) and 1.00 Å (aliphatic CH), and with $U_{\rm iso}$(H) values of $1.2U_{\rm eq}$(C).

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1891). Services for accessing these data are described at the back of the journal.

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