organic papers

Xiao-Bing Wang and Ling-Yi Kong*

Department of Natural Medicinal Chemistry, China Pharmaceutical University, Nanjing 210009, People's Republic of China

Correspondence e-mail: lykong@jlonline.com

Key indicators

Single-crystal X-ray study

$T = 298$ K

Mean σ(C–C) = 0.006 Å

R factor = 0.050

wR factor = 0.178

Data-to-parameter ratio = 13.5

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

(2E,2′E)-4,6-Dichloro-1,3-phenylene bis(3-phenylacrylate)

The title compound, $C_{24}H_{16}Cl_2O_4$, has non-crystallographic twofold rotational symmetry and the double bonds of the cinnamoyl groups adopt E configurations. Molecules are linked via C—H···O interactions, forming zigzag chains along the c axis.

Comment

The title compound, (I), is an unexpected product obtained in our ongoing study of the synthesis of flavones and we report its crystal structure here. The molecule has a non-crystallographic twofold rotation axis passing through atoms C2 and C5 of the central benzene ring (Fig. 1). The cinnamoyl groups including atoms O1/O2/C7–C15 and O3/O4/C16–C24 are almost planar, the mean deviations from their least-squares planes being 0.135 and 0.023 Å, respectively, and they form a dihedral angle of 51.06 (5)°. The double bonds of the cinnamoyl groups have E configurations.
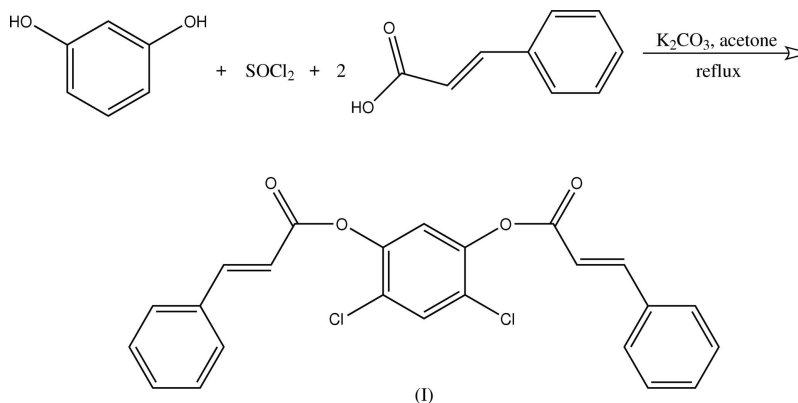
In the crystal structure, neighboring molecules are doubly linked via C—H···O interactions (Table 2), forming an $R_2^2(10)$ motif (Etter, 1990; Bernstein *et al.*, 1995; Desiraju, 1995). As a result, zigzag chains are formed along the c axis (Fig. 2).

Experimental

Cinnamic acid (3 g) was mixed with $SOCl_2$ under reflux. The mixture was added to an acetone solution of resorcinol (2 g) and anhydrous K_2CO_3 (6 g) with stirring, and refluxed for 12 h. The reaction mixture was concentrated for elimination of acetone. Ethyl acetate (50 ml) was added and the organic phase was washed three times with hot water, NaOH (5% aqueous solution) and hot water, respectively, dried over sodium sulfate and filtered. The mixture was then separated using flash column chromatography, yielding compound (I) as colorless crystals (yield 656 mg, 8%). Crystals of (I) suitable for X-ray

analysis were obtained by slow evaporation of an ethanol solution at room temperature.

Crystal data

$C_{24}H_{16}Cl_2O_4$
$M_r = 439.27$
Monoclinic, $P2_1/c$
$a = 10.829\ (2)$ Å
$b = 10.549\ (3)$ Å
$c = 18.368\ (4)$ Å
$\beta = 95.122\ (4)°$
$V = 2089.9\ (8)$ Å^3

$Z = 4$
$D_x = 1.396$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.34$ mm^{-1}
$T = 298\ (2)$ K
Prism, colorless
$0.42 \times 0.36 \times 0.24$ mm

Data collection

Bruker SMART 1000 CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2000)
$T_{min} = 0.871$, $T_{max} = 0.923$

10712 measured reflections
3659 independent reflections
1883 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.179$
$S = 1.01$
3659 reflections
271 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0761P)^2 + 1.169P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

Table 1
Selected torsion angles (°).

C16—O3—C4—C3	−86.7 (5)	C8—C9—C10—C11	9.8 (6)
C7—O1—C6—C5	88.7 (4)	O3—C16—C17—C18	177.9 (4)
O1—C7—C8—C9	−174.4 (3)	C16—C17—C18—C19	178.7 (4)
C7—C8—C9—C10	171.4 (3)	C17—C18—C19—C20	2.2 (7)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C9—H9$\cdots$O4^i	0.93	2.40	3.321 (5)	169
C18—H18$\cdots$O2^{ii}	0.93	2.43	3.295 (5)	155

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

All H atoms were positioned geometrically (C—H = 0.93 Å) and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve
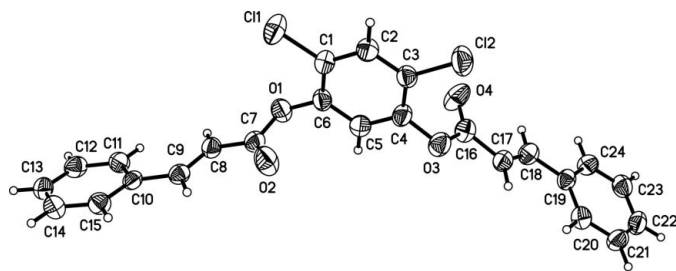

Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.
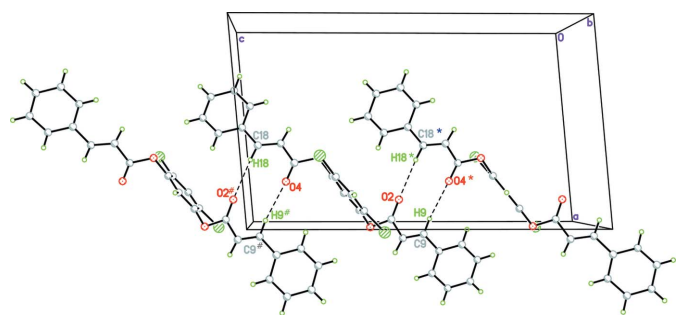

Figure 2
Part of the crystal structure of (I), showing a molecular chain formed via C—H$\cdots$O interactions (dashed lines). Atoms marked with an asterisk (*) and a hash (#) are at the symmetry positions $(x, \frac{1}{2} - y, z + \frac{1}{2})$ and $(x, \frac{1}{2} - y, z - \frac{1}{2})$, respectively.

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 and PLATON (Spek, 2003).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). Angew. Chem. Ed. Engl. **34**, 1555–1573.
Bruker (2000). SADABS, SAINT, SHELXTL and SMART. Bruker AXS Inc., Madison, Wisconsin, USA.
Desiraju, G. R. (1995). Angew. Chem. Int. Ed. Engl. **34**, 2311–2327.
Etter, M. C. (1990). Acc. Chem. Res. **23**, 120–126.
Sheldrick, G. M. (1997). SHELXL97 and SHELXS97. University of Göttingen, Germany.
Spek, A. L. (2003). J. Appl. Cryst. **36**, 7–13.