Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

N-[3-Phenyl-1-(2,3,5,6-tetrachloro-4-hydroxyphenoxy)prop-2-enylidene]aniline

Xiu-Rong Hu* and Jian-Ming Gu

Center of Analysis and Measurement, Zhejiang University, Hangzhou, Zhejiang 310028, People's Republic of China

Correspondence e-mail:
huxiurong@yahoo.com.cn

In the asymmetric unit of title structure, $C_{21}H_{13}Cl_4NO_2$, there are two independent molecules. Each independent molecule is linked to a symmetry-equivalent molecule by intermolecular O—H···N hydrogen bonds, forming a centrosymmetric dimer.

Received 3 August 2004
Accepted 9 September 2004
Online 18 September 2004

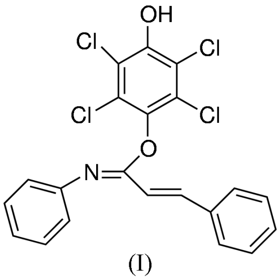
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.117
Data-to-parameter ratio = 13.9

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

Comment

Photo-induced reactions of halogenoquinones with electron donors such as alkenes, heterocycles, aromatics, hydrocarbons and amines are currently of interest (Maruyama & Imahori, 1990; Xu *et al.*, 1994). The photo-induced reaction of chloranil with a Schiff base (Chen *et al.*, 1994) provided the title compound, (I).



(I)

There are two independent molecules in the asymmetric unit of the title compound, as shown as Fig. 1. The two independent molecules have essentially the same conformation and the bond lengths and angles in each molecule are the same within experimental error, except for a slight difference in the

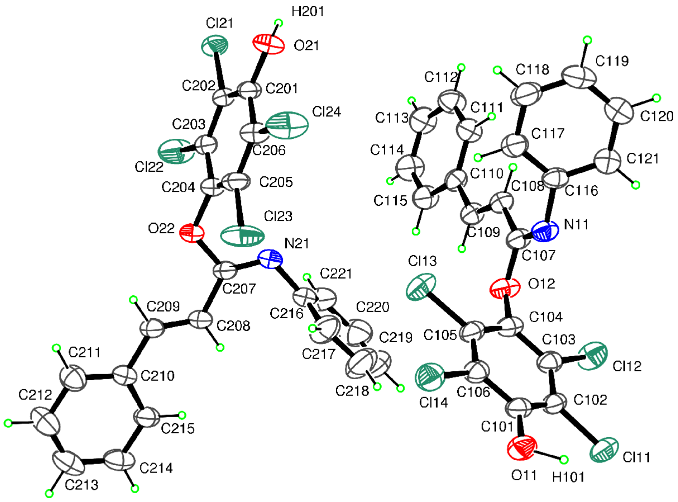


Figure 1
The two independent molecules of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

DOI: 10.1107/S1600536804022354

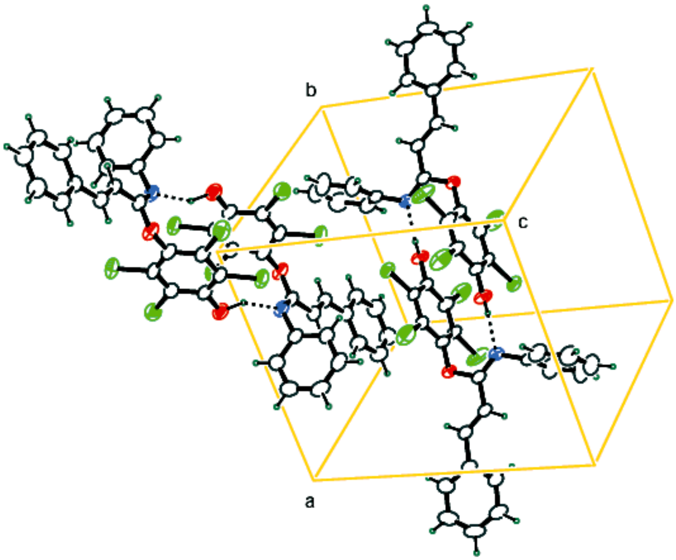


Figure 2
Part of the crystal structure of (I). Symmetry codes: (*) $-x, -y, -z$, (**) $1 - x, 1 - y, 1 - z$. Hydrogen bonds are shown as dashed lines.

magnitude of the angles C109—C110—C115 and C209—C210—C215 (see Table 1). In both molecules, the C═C double bond and imide group (C═N) display an *anti* conformation and are essentially planar with an *S-trans* conformation. This is illustrated clearly by the torsion angles N11—C107—C108—C109 [176.6 (3)°] and N21—C207—C208—C209 [179.2 (7)°] (Table 2 and Fig. 2).

Each independent molecule is linked into a centrosymmetric dimer by intermolecular O—H···N hydrogen bonds (Table 2). Hydroxy atoms O11 and O21 act as hydrogen-bond donors, *via* atoms H101 and H201 respectively, to imine atoms N11($-x, -y, -z$) and N21($1 - x, 1 - y, 1 - z$), respectively (Fig. 2).

Experimental

Photolysis of chloranil (0.05 mol l^{-1}) with *N*-cinnamylideneaniline (0.08 mol l^{-1}) in benzene with light of $\lambda > 400$ nm for 6.5 h gave a single product (Muler *et al.*, 1993), which was recrystallized from acetonitrile to obtain crystals suitable for X-ray diffraction.

Crystal data

$C_{21}H_{13}Cl_4NO_2$
$M_r = 453.15$
Triclinic, $P\overline{1}$
$a = 11.7423$ (4) Å
$b = 12.4902$ (6) Å
$c = 13.8929$ (4) Å
$\alpha = 95.577$ (3)°
$\beta = 91.2210$ (8)°
$\gamma = 90.524$ (2)°
$V = 2027.3$ (1) Å^3
$Z = 4$
$D_x = 1.485$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9200 reflections
$\theta = 1.5$–27.5°
$\mu = 0.60$ mm^{-1}
$T = 295$ (1) K
Chunk, colorless
0.70 × 0.60 × 0.25 mm

Data collection

Rigaku RAXIS–RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.678$, $T_{max} = 0.864$
15459 measured reflections
7043 independent reflections
6021 reflections with $F^2 > 2.0\sigma(F^2)$
$R_{int} = 0.019$
$\theta_{max} = 25.1$°
$h = -14 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.117$
$S = 1.10$
7043 reflections
508 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0591P)^2 + 0.7447P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl14—C106	1.723 (3)	C120—C121	1.374 (4)
Cl24—C206	1.710 (3)	C201—C206	1.388 (3)
C101—C102	1.392 (3)	C204—C205	1.376 (4)
C104—C105	1.387 (3)	C205—C206	1.395 (4)
C105—C106	1.383 (3)	C208—C209	1.319 (3)
C108—C109	1.330 (3)	C209—C210	1.465 (3)
C109—C110	1.455 (3)	C210—C211	1.382 (4)
C110—C111	1.405 (3)	C217—C218	1.383 (5)
C117—C118	1.392 (4)	C219—C220	1.365 (6)
C119—C120	1.373 (5)	C220—C221	1.385 (4)
C109—C110—C115	119.3 (2)	C209—C210—C215	122.9 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O11—H101···N11^{i}	1.06	1.90	2.818 (3)	143
O21—H201···N21^{ii}	1.01	1.83	2.751 (3)	149

Symmetry codes: (i) $-x, -y, -z$; (ii) $1 - x, 1 - y, 1 - z$.

Atoms H101 and H201 were located in difference Fourier maps and included in the refinement based on the as-found bond lengths, but riding on the O atoms to which they are bonded. Their isotropic displacement parameters were refined. The longer than normal O—H bond lengths are acceptable because of their involvement in intermolecular hydrogen bonding. All other H atoms were placed in calculated positions, with C—H = 0.98 Å, and refined in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}$(parent atom).

Data collection and cell refinement: *PROCESS-AUTO* (Rigaku, 1998); data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997).

References

Altomare, A., Burla, M., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Chen, C. F., Zhang, Z. G., Yan, B. Z. & Xu, J. H. (1994). *Tetrahedron Lett.* **35**, 9221–9224.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Maruyama, K. & Imahori, H. (1990). *J. Chem. Soc. Perkin Trans* 2, pp. 257–258.
Muler, F., Mattay, J. & Steenken, S. (1993). *J. Org. Chem.* **58**, 4462–4464.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, 3-9-12 Akishima, Tokyo 196-8666, Japan.
Rigaku/MSC (2004). *CrystalStructure*. Version 3.60. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381–5209, USA.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Xu, J. H., Song, Y. L., Zhang, Z. G., Wang, L. C. & Xu, J. W. (1994). *Tetrahedron*, **50**, 1199–1202.