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4-(4,5,6,7-Tetrachloro-1,3-dioxoisoindolin-2-yl)benzaldehyde

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The title compound, $C_{15}H_5Cl_4NO_3$, was synthesized from tetrachlorophthalic anhydride and 4-aminobenzaldehyde. In the molecule, the tetrachlorophthalimide unit is nearly planar and makes a dihedral angle of 56.20 (3)° with the benzene ring.

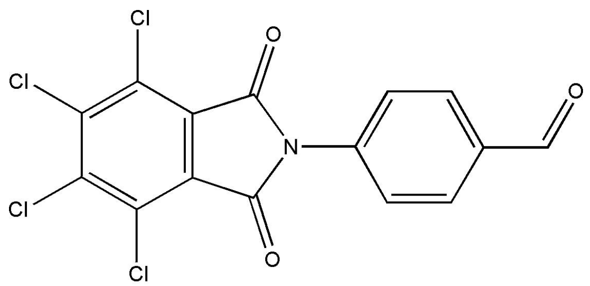
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.085
Data-to-parameter ratio = 17.4

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

Comment

Phthalimides and *N*-substituted phthalimides are an important class of compounds because of their interesting biological activities (Lima *et al.*, 2002; Orzeszka *et al.*, 2000). The photophysical properties of phthalimides have been studied intensively during the last two decades (Wintgens *et al.*, 1994; Griesbeck & Gomer, 1999; Hayashi *et al.*, 1980; Coyle *et al.*, 1978). Phthalimides have also served as starting materials and intermediates for the syntheses of alkaloids (Couture *et al.*, 1998) and pharmacophores (Couture *et al.*, 1997).



(I)

The bond lengths and angles in (I) have similar values to those in 4-(1,3-dioxoisoindolin-2-yl)benzaldehyde (DBA; Liu *et al.*, 2004). The tetrachlorophthalimide unit is nearly planar, with a mean deviation of 0.009 (2) Å for all contributing atoms. The dihedral angle between this plane and that of the

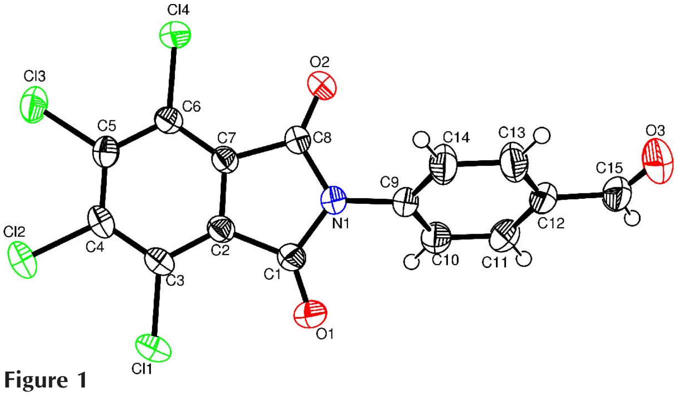


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms are drawn as spheres of arbitrary radius.

benzene ring of the benzaldehyde moiety is 56.20 (3)°. The Cl—C bond lengths in the substituted tetrachlorophthalimide unit are in agreement with values reported in the literature (Sutherland & Ali-Adib, 1987).

The occurrence of intermolecular C—H···O interactions results in the formation of discrete dimeric units (Fig. 2, Table 1).

Experimental

A solution of tetrachlorophthalic anhydride (10 mmol) and 4-aminobenzaldehyde (10 mmol) in acetic acid (40 ml) was heated at reflux until the disappearance of the starting materials, as evidenced by TLC. The resulting white precipitate was filtered off and washed with a minimal amount of water. The crude product was crystallized from acetic acid to give the title compound, (I). Recrystallization from acetone over 18 d at ambient temperature gave colorless single crystals of (I) suitable for X-ray analysis. ^{1}H NMR (CDCl$_3$, p.p.m.): 7.66 (*d*, J = 8.7 Hz, 2H), 8.03 (*d*, J = 8.4 Hz, 2H), 10.08 (*s* 1H).

Crystal data

$C_{15}H_5Cl_4NO_3$
$M_r = 389$
Monoclinic, $P2_1/c$
$a = 12.6686$ (10) Å
$b = 8.9057$ (7) Å
$c = 13.4400$ (11) Å
$\beta = 91.7370$ (10)°
$V = 1515.6$ (2) Å^3
$Z = 4$
$D_x = 1.705$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4015 reflections
$\theta = 2.7$–27.8°
$\mu = 0.79$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.28 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{\min} = 0.801$, $T_{\max} = 0.867$
9918 measured reflections
3619 independent reflections
2932 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$
$\theta_{\max} = 27.9°$
$h = -15 \rightarrow 16$
$k = -11 \rightarrow 11$
$l = -17 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.085$
$S = 1.10$
3619 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.0714P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···O1$^{\rm i}$	0.93	2.57	3.359 (2)	143.0

Symmetry code: (i) $1 - x, 1 - y, -z$.

H atoms were positioned geometrically (C—H = 0.93 Å) and refined as riding on their parent C atoms, with $U_{\rm iso}$ (H) = 1.2 $U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Bruker, 1997).

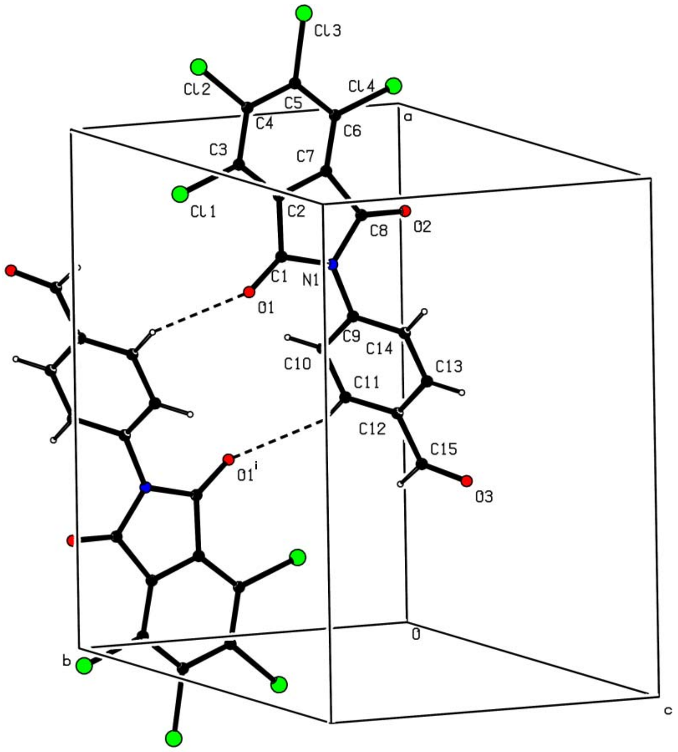


Figure 2
View of the dimer formed by C—H···O interactions (dashed lines). [Symmetry code: (i) $1 - x, 1 - y, -z$].

References

Bruker (1997). *SADABS*, *SMART*, *SAINT* and *SHELXTL*.. Bruker AXS Inc., Madison, Wisconsin, USA.

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA.

Couture, A., Deniau, E., Grandclaudon, P. & Hoarau, C. (1998). *J. Org. Chem.* **63**, 3128–3132.

Couture, A., Deniau, E., Woisel, P. & Grandclaudon, P. (1997). *Synthesis*, **63**, 1469–1445.

Coyle, J. D., Newport, G. L. & Harriman, A. (1978). *J. Chem. Soc. Perkin Trans* 2, pp. 133–137.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Griesbeck, A. G. & Gomer, H. (1999). *J. Photochem. Photobiol. A Chem.* **129**, 111–119.

Hayashi, H., Nagakura, S., Kubo, Y. & Maruyama, K. (1980). *Chem. Phys. Lett.* **72**, 291–294.

Lima, L. M., Castro, P., Machado, A. L., Frage, C. A. M., Lugniur, C., Moraes, V. L. G. & Barreiro, E. (2002). *J. Bio. Org. Med. Chem.* **10**, 3067–3073.

Liu, X. G., Feng, Y. Q., Wu, P., Chen, X. & Li, F. (2004). *Acta Cryst.* E**60**, o2293–o2294.

Orzeszka, A., Kaminsaka, B., Orzesko, G. & Stareerciak, J. (2000). *Farmaco*, **55**, 619–623.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Sutherland, H. H. & Ali-Adib, Z. (1987). *Acta Cryst.* C**36**, 1406–1407.

Wintgens, V., Valat, P., Kossanyi, J., Biczok, L., Demeter, A. & Berces, T. (1994). *J. Chem. Soc. Faraday Trans.* **90**, 411–421.

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