Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(3-Chloropropyl)-2,3-dihydro-1*H*-isoindole-1,3-dione

Basavegowda Nagaraj,[a] Hemmige S. Yathirajan[a] and Michael Bolte[b]*

[a]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, and [b]Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail: bolte@chemie.uni-frankfurt.de

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.029
wR factor = 0.074
Data-to-parameter ratio = 16.7

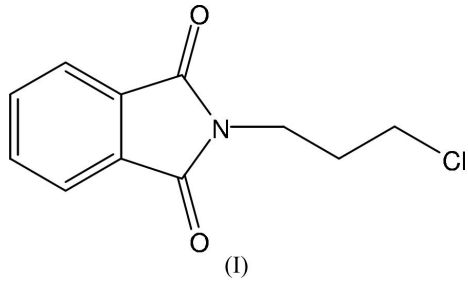
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 February 2005
Accepted 2 March 2005
Online 11 March 2005

The geometric parameters of the title compound, $C_{11}H_{10}ClNO_2$, are in the normal ranges. The phthalimide moiety is planar and the chloropropyl chain adopts a synclinal conformation. The crystal packing is stabilized by two intermolecular C—H···O contacts.

Comment

The title compound, (I), also named *N*-(3-chloropropyl)-phthalimide, is used as an intermediate for the synthesis of biologically active heterocycles (Kerrigan *et al.*, 2000; Salvati *et al.*, 2005). In view of its importance and in order to determine the conformation of this molecule, a crystal structure determination has been carried out.



(I)

A perspective view of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal [Cambridge Structural Database (CSD), Version 1.7; *MOGUL*, Version 1.0.1; Allen, 2002]. The isoindole-1,3-dione system is planar (r.m.s. deviation = 0.011 Å). Methylene atom C9 attached to the N atom deviates from this plane by only 0.031 (1) Å. The chloropropyl moiety adopts a synclinal conformation [Cl1—C11—C10—C9 = −67.71 (12)°]. This conformation is also found for seven out of eight structures containing the Cl—$CH_2CH_2CH_2$—*AA* fragment (*AA* = any atom) retrieved from the CSD. The only

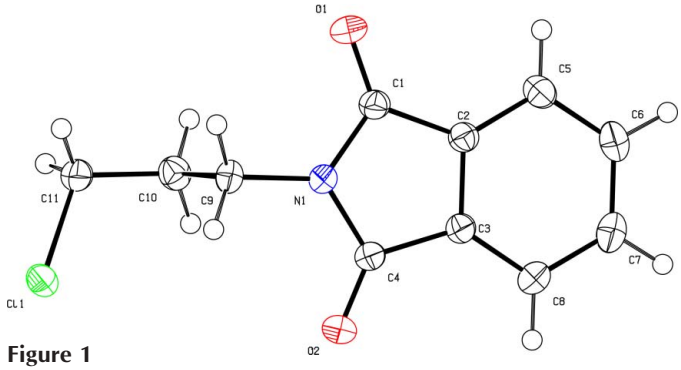


Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level.

© 2005 International Union of Crystallography
Printed in Great Britain – all rights reserved

structure not showing this conformation is 8-amino-7-(3-chloropropyl)theophylline benzene solvate, with a torsion angle of 175.8° (Karczmarzyk & Pawlowski, 1998). The —$CH_2CH_2CH_2$—N chain, on the other hand, adopts an anti-periplanar conformation [C11—C10—C9—N1 = 177.15 (10)°]. The crystal packing is stabilized by two intermolecular C—H···O contacts (Table 2).

Experimental

A mixture of isoindole-1,3-dione (1.47 g, 10 mmol), anhydrous potassium carbonate (1.38 g, 10 mmol) and 1-bromo-3-chloropropane (1.57 g, 10 mmol) was stirred at room temperature in dimethylformamide (10 ml) for 6 h to give the title compound, which was recrystallized from methanol (m.p. 340 K).

Crystal data

$C_{11}H_{10}ClNO_2$
M_r = 223.65
Monoclinic, $P2_1/n$
a = 4.5421 (4) Å
b = 15.3996 (15) Å
c = 15.3471 (13) Å
β = 96.605 (7)°
V = 1066.35 (17) Å^3
Z = 4
D_x = 1.393 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 29 481 reflections
θ = 3.7–27.1°
μ = 0.34 mm^{-1}
T = 173 (2) K
Rod, colourless
0.38 × 0.22 × 0.22 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.883, T_{max} = 0.930
11 334 measured reflections
2290 independent reflections
2103 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 26.9°
$h = -5 \rightarrow 5$
$k = -19 \rightarrow 19$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.074
S = 1.06
2290 reflections
137 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 0.3497P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.064 (5)

Table 1
Selected bond lengths (Å).

N1—C1	1.4001 (15)	C1—O1	1.2165 (14)
N1—C4	1.4015 (14)	C4—O2	1.2183 (15)
N1—C9	1.4660 (14)	C11—Cl1	1.8198 (13)

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11A···O2^{i}	0.99	2.57	3.3251 (15)	133
C11—H11B···O1^{ii}	0.99	2.43	3.2494 (15)	140

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

All H atoms were located in a difference map, but were then geometrically positioned and refined with fixed individual displacement parameters (set at 1.2 times U_{eq} of the parent atom) using a riding model, with C—H = 0.95 and 0.99 Å for aromatic and methylene H atoms, respectively.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Karczmarzyk, Z. & Pawlowski, M. (1998). *Pol. J. Chem.* **72**, 793–798.
Kerrigan, J. E., Walters, M. C., Forrester, K. J., Crowder, J. B. & Christopher, L. J. (2000). *Bioorg. Med. Chem. Lett.* **10**, 27–30.
Salvati, M. E., Balog, A., Wei, D. D., Pickering, D., Attar, R. M., Geng, J., Rizzo, C. A., Hunt, J. T., Gottardis, M. M., Weinmann, R. & Martinez, R. (2005). *Bioorg. Med. Chem. Lett.* **15**, 389–393.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.