

Table 2. Selected geometric parameters (Å, °)

|                      |           |                      |           |
|----------------------|-----------|----------------------|-----------|
| O(1)—C(1)            | 1.404 (5) | O(6)—C(6)            | 1.379 (4) |
| O(1)—C(10)           | 1.433 (4) | O(6)—C(7)            | 1.404 (4) |
| O(2)—C(2)            | 1.427 (4) | C(1)—C(2)            | 1.536 (4) |
| O(2)—C(10)           | 1.419 (4) | C(2)—C(3)            | 1.521 (4) |
| O(4)—C(1)            | 1.411 (3) | C(3)—C(4)            | 1.533 (4) |
| O(4)—C(4)            | 1.441 (3) | C(3)—C(13)           | 1.527 (3) |
| O(5)—C(5)            | 1.433 (4) | C(4)—C(5)            | 1.511 (4) |
| O(5)—C(7)            | 1.422 (3) | C(5)—C(6)            | 1.522 (5) |
| C(1)—O(1)—C(10)      | 109.8 (3) | O(2)—C(2)—C(3)       | 109.4 (2) |
| C(2)—O(2)—C(10)      | 107.8 (2) | C(1)—C(2)—C(3)       | 104.6 (2) |
| C(1)—O(4)—C(4)       | 109.0 (2) | C(2)—C(3)—C(4)       | 100.8 (2) |
| C(5)—O(5)—C(7)       | 107.8 (2) | O(4)—C(4)—C(3)       | 104.0 (2) |
| C(6)—O(6)—C(7)       | 110.7 (3) | O(5)—C(5)—C(6)       | 102.8 (2) |
| O(1)—C(1)—O(4)       | 111.8 (3) | O(6)—C(6)—C(5)       | 104.0 (3) |
| O(1)—C(1)—C(2)       | 105.3 (2) | O(5)—C(7)—O(6)       | 106.9 (2) |
| O(4)—C(1)—C(2)       | 106.8 (2) | O(1)—C(10)—O(2)      | 104.6 (3) |
| O(2)—C(2)—C(1)       | 103.0 (3) |                      |           |
| O(1)—C(1)—C(2)—O(2)  | -15.4 (3) | C(1)—O(4)—C(4)—C(3)  | 33.6 (3)  |
| O(1)—C(10)—O(2)—C(2) | -31.3 (3) | C(1)—C(2)—O(2)—C(10) | 28.8 (3)  |
| O(2)—C(10)—O(1)—C(1) | 20.9 (4)  | C(1)—C(2)—C(3)—C(4)  | 29.3 (3)  |
| O(4)—C(1)—C(2)—C(3)  | -10.7 (3) | C(2)—C(1)—O(1)—C(10) | -3.2 (3)  |
| O(4)—C(4)—C(3)—C(2)  | -38.2 (3) | C(2)—C(1)—O(4)—C(4)  | -14.3 (3) |
| O(5)—C(5)—C(6)—O(6)  | 27.7 (4)  | C(5)—C(6)—O(6)—C(7)  | -21.0 (5) |
| O(5)—C(7)—O(6)—C(6)  | 6.0 (5)   | C(6)—C(5)—O(5)—C(7)  | -24.7 (4) |
| O(6)—C(7)—O(5)—C(5)  | 13.0 (4)  |                      |           |

The H atoms were initially located in a difference electron density map, but their positions were subsequently geometrically idealized [ $d(\text{C—H}) = 0.95 \text{ \AA}$ ] and only their  $U$ 's were refined.

*MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991) was used for data collection and cell determination, and *TEXSAN* software (Molecular Structure Corporation, 1989) was used for data reduction, structure refinement and the preparation of publication material. The structure was solved using direct methods (*SHELXS86*; Sheldrick, 1990) and molecular graphics were produced using *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SX1012). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1996). **C52**, 1799–1801

## 2-Chlorobenzimidazole†

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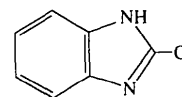
(Received 23 November 1994; accepted 22 December 1995)

### Abstract

The crystal structure of the title compound,  $\text{C}_7\text{H}_5\text{ClN}_2$ , is stabilized by an  $\text{N—H}\cdots\text{N}$  hydrogen bond,  $\text{C—H}\cdots\text{Cl}$  non-bonded intermolecular interactions and a  $\text{halogen}\cdots\text{halogen}$  interaction.

### Comment

The crystal and molecular structure of the title compound, (1), was investigated in order to determine the conformation and packing.



(1)

Bond distances and angles are in agreement with a related structure reported by Sprang & Sundaralingam (1973), *i.e.* 2-chloro-1-( $\beta$ -D-ribofuranosyl)benzimidazole. The average bond distance in the phenyl ring is  $1.383(6) \text{ \AA}$  and the molecule is planar within  $0.004(2) \text{ \AA}$  (Fig. 1). The H atom at the N9 position participates in an  $\text{N—H}\cdots\text{N}$  hydrogen bond with the symmetry-related  $\text{N7}^i$  atom at a distance of  $2.959(5) \text{ \AA}$  [ $\text{H9}\cdots\text{N7}^i$   $2.05(6) \text{ \AA}$  and  $\text{N9—H9}\cdots\text{N7}^i$   $172(5)^\circ$ ; symmetry code: (i)  $-\frac{1}{2} + x, \frac{3}{2} - y, 1 - z$ ] (Desiraju, 1991). The C2 atom forms a non-bonded interaction with the  $\text{Cl1}^i$  atom [ $\text{C2}\cdots\text{Cl1}^i$   $3.778(5)$ ,  $\text{H2}\cdots\text{Cl1}^i$   $3.04(4) \text{ \AA}$  and  $\text{C2—H2}\cdots\text{Cl1}^i$   $149(4)^\circ$ ]. Also, the C5 atom is involved in a  $\text{C—H}\cdots\text{Cl}$  non-bonded interaction with the  $\text{Cl1}^{ii}$  atom [ $\text{C5}\cdots\text{Cl1}^{ii}$   $3.771(5)$ ,  $\text{H5}\cdots\text{Cl1}^{ii}$   $2.86(4) \text{ \AA}$  and  $\text{C5—H5}\cdots\text{Cl1}^{ii}$   $155(3)^\circ$ ; symmetry code: (ii)  $\frac{1}{2} + x$ ,

† Contribution No. 1423 of the Instituto de Química, UNAM.

$\frac{3}{2} - y, 1 - z$ ]. The structure in the crystal shows a pattern in which two molecules are linked through a pair of non-bonded C—H...Cl interactions and an N—H...N hydrogen bond (Fig. 2). There is also one halogen...halogen non-bonded interaction (Desiraju, 1989) [Cl1...Cl1<sup>iii</sup> 3.841 (2) Å; symmetry code: (iii)  $1 - x, 2 - y, 1 - z$ ].

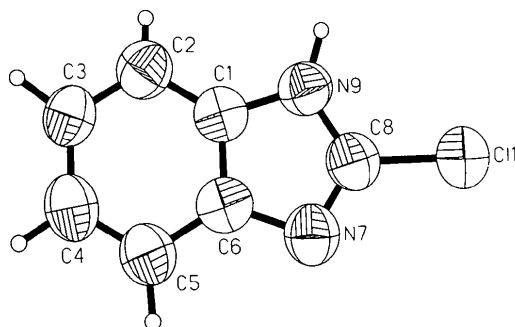


Fig. 1. The molecular structure of the title compound with the atom labelling and 50% probability displacement ellipsoids.

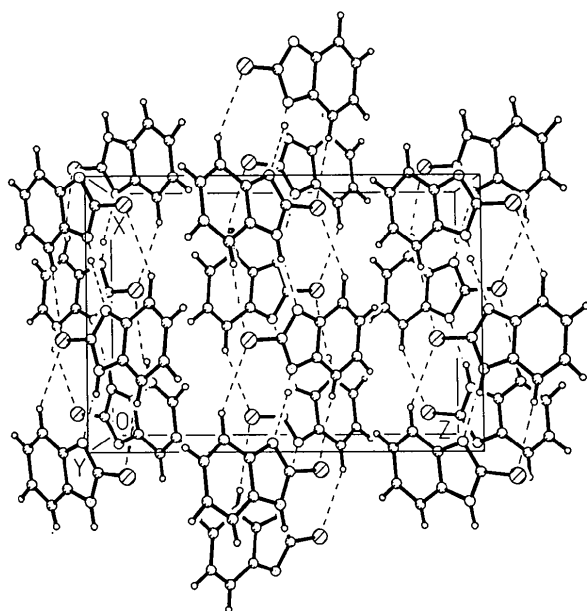


Fig. 2. A perspective view of the packing arrangement in the title crystal, with dashed lines indicating C—H...Cl non-bonded intermolecular interactions and N—H...N hydrogen bonding.

## Experimental

The crystal of (1) used for analysis was obtained from an acetone solution of the commercially available material by slow evaporation of the solvent.

### Crystal data

C<sub>7</sub>H<sub>5</sub>ClN<sub>2</sub>  
M<sub>r</sub> = 152.58

Cu Kα radiation  
λ = 1.54178 Å

Orthorhombic

Pbca

a = 9.937 (4) Å

b = 9.192 (1) Å

c = 14.417 (3) Å

V = 1316.9 (6) Å<sup>3</sup>

Z = 8

D<sub>r</sub> = 1.539 Mg m<sup>-3</sup>

Cell parameters from 25 reflections

θ = 15–45°

μ = 4.385 mm<sup>-1</sup>

T = 293 (2) K

Rectangular

0.30 × 0.25 × 0.22 mm

Colourless

### Data collection

P4 diffractometer

θ/2θ scans

Absorption correction:

semi-empirical via ψ

scan (North, Phillips &

Mathews, 1968)

T<sub>min</sub> = 0.067, T<sub>max</sub> =

0.496

779 measured reflections

779 independent reflections

717 observed reflections

[I > 2σ(I)]

θ<sub>max</sub> = 56.74°

h = 0 → 10

k = 0 → 9

l = 0 → 15

3 standard reflections

monitored every 100

reflections

intensity decay: 2%

### Refinement

Refinement on F<sup>2</sup>

R(F) = 0.0482

wR(F<sup>2</sup>) = 0.1736

S = 1.231

776 reflections

111 parameters

All H atoms are refined

isotropically

w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.0732P)<sup>2</sup>

+ 0.4705P]

where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3

(Δ/σ)<sub>max</sub> = -0.004

Δρ<sub>max</sub> = 0.175 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.131 e Å<sup>-3</sup>

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

|     | x          | y          | z          | U <sub>eq</sub> |
|-----|------------|------------|------------|-----------------|
| Cl1 | 0.4035 (1) | 0.8919 (1) | 0.4074 (1) | 0.0932 (6)      |
| C1  | 0.3336 (4) | 0.5947 (4) | 0.5855 (2) | 0.0717 (10)     |
| C2  | 0.2630 (5) | 0.4948 (4) | 0.6377 (3) | 0.0816 (11)     |
| C3  | 0.3337 (4) | 0.4102 (4) | 0.6985 (2) | 0.0824 (11)     |
| C4  | 0.4731 (5) | 0.4262 (4) | 0.7083 (2) | 0.0862 (11)     |
| C5  | 0.5434 (4) | 0.5276 (4) | 0.6574 (2) | 0.0820 (11)     |
| C6  | 0.4715 (4) | 0.6126 (4) | 0.5948 (2) | 0.0731 (10)     |
| N7  | 0.5175 (3) | 0.7213 (3) | 0.5343 (2) | 0.0797 (9)      |
| C8  | 0.4081 (3) | 0.7607 (4) | 0.4920 (3) | 0.0752 (10)     |
| N9  | 0.2945 (3) | 0.6925 (3) | 0.5173 (2) | 0.0748 (9)      |

Table 2. Selected geometric parameters (Å, °)

|          |           |           |           |
|----------|-----------|-----------|-----------|
| C1—C8    | 1.715 (4) | C4—C5     | 1.376 (5) |
| C1—C2    | 1.379 (5) | C5—C6     | 1.392 (5) |
| C1—C6    | 1.386 (6) | C6—N7     | 1.403 (4) |
| C1—N9    | 1.388 (5) | N7—C8     | 1.298 (4) |
| C2—C3    | 1.366 (6) | C8—N9     | 1.342 (5) |
| C3—C4    | 1.400 (7) |           |           |
| C2—C1—C6 | 122.0 (3) | C1—C6—N7  | 110.3 (3) |
| C2—C1—N9 | 132.5 (4) | C5—C6—N7  | 129.5 (4) |
| C6—C1—N9 | 105.6 (3) | C8—N7—C6  | 102.6 (3) |
| C3—C2—C1 | 117.9 (4) | N7—C8—N9  | 116.5 (3) |
| C2—C3—C4 | 120.9 (4) | N7—C8—C11 | 123.5 (3) |
| C5—C4—C3 | 121.3 (4) | N9—C8—C11 | 120.0 (3) |
| C4—C5—C6 | 117.7 (4) | C8—N9—C1  | 105.0 (3) |
| C1—C6—C5 | 120.2 (3) |           |           |

Data collection: *P4* diffractometer software. Cell refinement: *XSCANS* (Siemens, 1991). Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL93*.

KP acknowledges the CONACYT (Cátedra Patrimonial Nivel II) for fellowship assistance. The authors thank the Instituto de Biotecnología, UNAM, for X-ray data collection.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SZ1041). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1996). **C52**, 1801–1804

## 2,4,6-Trichlorobenzoic Acid: Structure and Hydrogen-Bonding Pattern

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(Received 29 June 1995; accepted 30 January 1996)

### Abstract

This is an uncommon structure with  $Z = 12$  in space group  $P2_1/n$ . The three molecules of  $C_7H_3Cl_3O_2$  in the asymmetric unit consist of one complete carboxyl-

ic acid dimer at a general position and one-half of a second unique dimer located at a crystallographic inversion center. Each of the three acid molecules in the asymmetric unit displays carboxyl bond lengths and angles that are nearly equivalent to each other (as opposed to the highly defined C=O and C—O distances, and C—C—O angles that occur in very ordered dimeric systems).

### Comment

We have been interested for some time in the mode of hydrogen bonding in simple carboxylic acids, mainly those containing a ketone as a second functional group. When the ketone functional group is present, there are four different types of hydrogen-bonding states: dimerization is by far the most common hydrogen-bonding motif, the next is the catemer (intermolecular carboxyl-to-ketone), then there are also the rarer forms of internal hydrogen bond and one instance of acid-to-ketone dimerization (Thompson, Lalancette & Vanderhoff, 1992; Coté, Thompson & Lalancette, 1996). When the second functional group (ketone) is not present, the acid group can present itself as a dimer (Miller, Paul & Curtin, 1974; Takazawa, Ohba & Saito, 1989; Takwale & Pant, 1971; Ferguson & Sim, 1961), or as a catemer like acetic acid (Jones & Templeton, 1958; Nahrungbauer, 1970) and formic acid (Holtzberg, Post & Fankuchen, 1953; Nahrungbauer, 1978).

The electron-withdrawing power of the three Cl atoms in the title compound, (I), led us to believe that we might be able to crystallize this compound with a molecule of solvent inserted between the dimers, such as the two water molecules that expand the eight-membered dimeric structure of anhydrous 2-benzoylbenzoic acid to the hydrated form of the acid having a 12-membered ring structure (Lalancette, Vanderhoff & Thompson, 1990). The structure of pentafluorobenzoic acid (Benghiat & Leiserowitz, 1972) shows that this molecule adopts the dimeric conformation when it is crystallized from non-aqueous solvents. The electron-withdrawing power of the five F atoms should allow for the incorporation of a solvent molecule between the dimers; however, this experiment has not yet been reported in the literature. In addition, the present structure also shows no evidence of molecules of solvation.

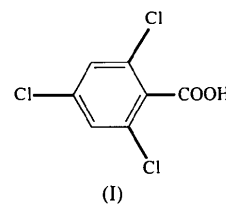


Fig. 1 presents a view of the three independent molecules of (I) in the asymmetric unit ( $Z = 12$ ) with