Data collection: *P*4 diffractometer software. Cell refinement: *XSCANS* (Siemens, 1991). Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL*93.

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

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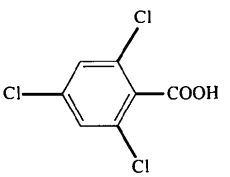
Abstract

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 carboxylic 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; Nahringbauer, 1970) and formic acid (Holtzberg, Post & Fankuchen, 1953; Nahringbauer, 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.



(I)

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

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the atomic numbering scheme. Also shown is a fourth molecule (the centrosymmetrically related dimer molecule of *A* drawn with open anisotropic atoms and open bonds, for clarity). Molecule *A* is situated at the crystallographic center of symmetry, while molecules *B* and *C* are two independent molecules in general positions.

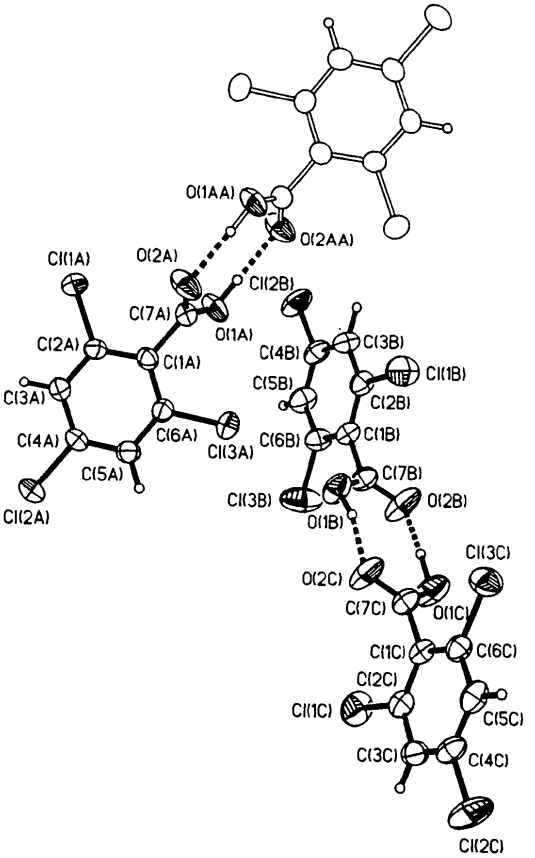

Fig. 1. A view of the three molecules of the asymmetric unit (with the centrosymmetrically related dimer molecule to *A* drawn with open ellipsoids and open bonds for clarity). The carboxyl groups are all partially disordered and the carboxyl H atoms were fixed 0.98 Å from their respective O atoms. Ellipsoids are drawn at the 40% probability level.

None of the three molecules is planar. The plane of the carboxyl group [C(7)—O(1)—O(2)—H(1)] in each molecule is twisted out of the plane of the trichlorobenzene ring, [Cl(1)—Cl(2)—Cl(3) and C(1) through C(6)], by 76.2 (5)° in molecule *A* (the crystallographic dimer), by 83.5 (1)° in molecule *B*, and by 87.1 (5)° in molecule *C*. Additionally, the relationship of the trichlorobenzenoid rings of the two different dimers is of interest: for molecule *A*, these two ring systems are coplanar. However, the rings making up molecules *B* and *C* deviate from coplanarity by 7.9 (1)°.

Disordering in the carboxyl group is displayed by the C—O bond length and C—C—O angles. This disordering is common in dimeric acids (Dieterich, Paul & Curtin, 1974). The C—O bond lengths found in the title compound are 1.234 (6) and 1.269 (6) Å in molecule *A*, 1.233 (6) and 1.250 (6) Å in molecule *B*, and 1.241 (6) and 1.244 (6) Å in molecule *C*. The C—C—O angles found are 116.7 (4) and 118.2 (4)° in molecule *A*, 117.3 (4) and 117.8 (4)° in molecule *B*, and 117.8 (4)° for both of the angles in molecule *C*. These can be compared with typical lengths of 1.21 (3) Å for the C=O and 1.31 (2) Å for the C—O(H) bond distances, and angles of 112 (2)° for C—C—O(H) and 123 (2)° for C—C=O in some of the more highly ordered carboxyl dimers (Borthwick, 1980). There is essentially no rotational disorder about the C(1)—C(7) bond in these molecules.

As a result of the obvious disorder, it was difficult to find the carboxyl H atoms in difference maps. Once located, each H atom was constrained to be 0.98 Å from its respective O atom. The O···O distances in the centrosymmetric dimer are 2.664 (3) Å, but the distances between the O atoms in the non-crystallographic dimer (molecules *B* and *C*) are 2.661 (3) and 2.693 (3) Å, respectively. These are all normal O···O distances for carboxylic acid dimer pairs and are compared with 2.604 Å (Coté, Lalancette & Thompson, 1995) through 2.770 Å (Lalancette, Vanderhoff & Thompson, 1991). The angle O(1*A*)—H(1*A*)···O(2*A*^{i}) [(i) = $1-x$, $2-y$, $-z$] is 172 (3)° for the centrosymmetric dimer; O(1*B*)—H(1*B*)···O(2*C*) is 166 (3)° and O(1*C*)—H(1*C*)···O(2*B*) is 176 (3)° for the non-crystallographic dimers. These angles are typical for carboxylic acid dimers.

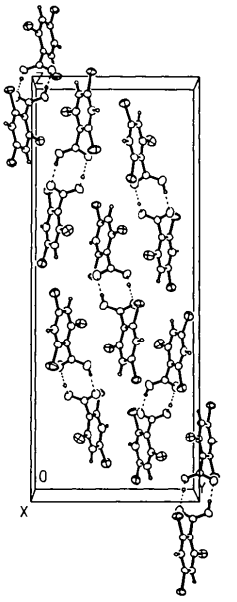

Fig. 2. A packing diagram for (I). The centrosymmetrically related molecules are shown at the center of the *bc* face and along cell edge *a*. The non-centrosymmetric dimer pairs are easily discerned in general positions. The Cl atoms are characterized with boundary and principal ellipses, and all of the ellipsoids are drawn at the 30% probability level.

A packing diagram of the cell (Fig. 2) displays the 12 molecules in the four asymmetric units in this structure. The centrosymmetrically related molecules are shown at the center of the *bc* face and along cell edge *a*. The non-centrosymmetric carboxylic acid pairs are easily discerned in their general positions. The closest intermolecular contact (aside from the acid dimer) is between the Cl atom [Cl(2)] (*para* to the acid group) and its counterpart in an adjacent molecule; this is 3.437 (2) Å. The closest perpendicular ring-to-ring 'stacking' distance is calculated to be 3.510 (3) Å; this is shorter than, but comparable to, the 'stacking' distance in the structure of 2,4,6-tribromoaniline (Christensen & Strömme, 1969), which we calculated to be 3.692 Å.

The volume of the cell seems somewhat large for the number of atoms encompassed within it. The calculated volume swept per atom in the cell is 14.9 Å^3. However, this seemingly large value compares favorably with those in the following multi-halogenated structures: 2-amino-3,5,6-trichloro-1,4-benzoquinone (Endres, Rossato & Balles, 1984) in which the volume swept per atom is 14.6 Å^3; 2,4,6-tribromoaniline (Christensen & Strömme, 1969) with a value of 15.0 Å^3; and tribromobenzene (Milledge & Pant, 1960) with a value of 16.4 Å^3.

The solid-state (KBr) IR spectrum of the compound displays a single, somewhat broadened C=O stretching absorption centered at 1720 cm^{-1}, which is normal for the carboxyl asymmetric stretch in a dimeric hydrogen-bonding motif (Vanderhoff, Lalancette & Thompson, 1990; Coté, Lalancette & Thompson, 1995).

Experimental

(I) was purchased from Aldrich Chemical Co., Inc., Milwaukee, Wisconsin, and crystallized by slow evaporation from ethyl butyl ketone at room temperature.

Crystal data

$C_7H_3Cl_3O_2$
M_r = 225.5
Monoclinic
$P2_1/n$
a = 7.259 (1) Å
b = 12.204 (2) Å
c = 30.275 (3) Å
β = 90.779 (9)°
V = 2681.7 (6) Å^3
Z = 12
D_x = 1.676 Mg m^{-3}
D_m = 1.674 (2) Mg m^{-3}
D_m measured by flotation in cyclohexane/$CBrCl_3$

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 43 reflections
θ = 11.1–17.7°
μ = 0.98 mm^{-1}
T = 296 K
Trapezoid
0.44 × 0.28 × 0.24 mm
Colorless

Data collection

Siemens *P*4 diffractometer
$2\theta/\omega$ scans

R_{int} = 0.0457
θ_{max} = 27.5°
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 39$
3 standard reflections monitored every 97 reflections
intensity decay: 5.53%

Absorption correction: face-indexed numerical
T_{min} = 0.637, T_{max} = 0.799
8305 measured reflections
6172 independent reflections
2551 observed reflections [$F > 4\sigma(F)$]

Refinement

Refinement on F
R = 0.045
wR = 0.047
S = 1.03
2551 reflections
336 parameters
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{max}$ = 0.130
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Extinction correction: $F^* = F/[1.0 + 0.002\chi F^2/\sin(2\theta)]^{1/4}$
Extinction coefficient: χ = 0.0006 (1)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cl(1A)	−0.0184 (2)	1.1321 (1)	0.0321 (1)	0.071 (1)
Cl(2A)	−0.1281 (2)	1.1147 (1)	0.2062 (1)	0.064 (1)
Cl(3A)	0.5036 (2)	0.9620 (1)	0.1407 (1)	0.066 (1)
O(1A)	0.3647 (5)	0.9321 (3)	0.0372 (1)	0.062 (1)
O(2A)	0.4356 (5)	1.1089 (3)	0.0341 (1)	0.075 (1)
C(1A)	0.2335 (6)	1.0497 (3)	0.0895 (1)	0.040 (1)
C(2A)	0.0612 (6)	1.0983 (4)	0.0846 (1)	0.041 (2)
C(3A)	−0.0520 (6)	1.1158 (4)	0.1195 (1)	0.046 (2)
C(4A)	0.0111 (6)	1.0887 (4)	0.1612 (1)	0.043 (2)
C(5A)	0.1830 (6)	1.0414 (4)	0.1683 (1)	0.047 (2)
C(6A)	0.2907 (6)	1.0218 (4)	0.1318 (1)	0.043 (2)
C(7A)	0.3536 (6)	1.0273 (4)	0.0507 (1)	0.044 (2)
Cl(1B)	1.0222 (2)	0.7936 (1)	0.0829 (1)	0.083 (1)
Cl(2B)	0.4016 (2)	0.6425 (1)	0.0071 (1)	0.086 (1)
Cl(3B)	0.4849 (3)	0.6259 (2)	0.1829 (1)	0.108 (1)
O(1B)	0.8394 (5)	0.8320 (3)	0.1846 (1)	0.071 (1)
O(2B)	0.9506 (7)	0.6643 (3)	0.1875 (1)	0.100 (2)
C(1B)	0.7427 (7)	0.7090 (4)	0.1296 (1)	0.045 (2)
C(2B)	0.8056 (6)	0.7351 (4)	0.0878 (2)	0.050 (2)
C(3B)	0.7039 (7)	0.7134 (4)	0.0502 (1)	0.055 (2)
C(4B)	0.5338 (8)	0.6661 (4)	0.0543 (2)	0.054 (2)
C(5B)	0.4642 (7)	0.6386 (4)	0.0947 (2)	0.060 (2)
C(6B)	0.5699 (7)	0.6600 (4)	0.1317 (1)	0.054 (2)
C(7B)	0.8523 (7)	0.7375 (4)	0.1705 (2)	0.051 (2)
Cl(1C)	0.9523 (2)	0.7830 (1)	0.3616 (1)	0.087 (1)
Cl(2C)	1.5628 (3)	0.9493 (1)	0.4359 (1)	0.101 (1)
Cl(3C)	1.5136 (2)	0.9082 (1)	0.2598 (1)	0.078 (1)
O(1C)	1.1441 (6)	0.7168 (3)	0.2613 (1)	0.085 (2)
O(2C)	1.0395 (6)	0.8854 (3)	0.2558 (1)	0.086 (2)
C(1C)	1.2434 (7)	0.8449 (4)	0.3146 (1)	0.045 (2)
C(2C)	1.1717 (7)	0.8338 (4)	0.3561 (2)	0.050 (2)
C(3C)	1.2670 (7)	0.8645 (4)	0.3939 (2)	0.056 (2)
C(4C)	1.4395 (8)	0.9083 (4)	0.3890 (2)	0.056 (2)
C(5C)	1.5183 (7)	0.9220 (4)	0.3481 (2)	0.058 (2)
C(6C)	1.4193 (7)	0.8896 (4)	0.3114 (1)	0.049 (2)
C(7C)	1.1339 (7)	0.8134 (4)	0.2742 (1)	0.052 (2)

Table 2. *Selected geometric parameters (Å, °)*

Cl(1A)—C(2A)	1.734 (4)	C(2B)—C(3B)	1.374 (6)
Cl(2A)—C(4A)	1.737 (4)	C(3B)—C(4B)	1.370 (7)
Cl(3A)—C(6A)	1.727 (5)	C(4B)—C(5B)	1.371 (7)
O(1A)—C(7A)	1.234 (6)	C(5B)—C(6B)	1.376 (7)

O(2A)—C(7A)	1.269 (6)	Cl(1C)—C(2C)	1.720 (5)
C(1A)—C(2A)	1.391 (6)	Cl(2C)—C(4C)	1.740 (5)
C(1A)—C(6A)	1.381 (6)	Cl(3C)—C(6C)	1.729 (5)
C(1A)—C(7A)	1.498 (6)	O(1C)—C(7C)	1.244 (6)
C(2A)—C(3A)	1.364 (6)	O(2C)—C(7C)	1.241 (6)
C(3A)—C(4A)	1.377 (6)	C(1C)—C(2C)	1.371 (6)
C(4A)—C(5A)	1.389 (6)	C(1C)—C(6C)	1.393 (7)
C(5A)—C(6A)	1.385 (6)	C(1C)—C(7C)	1.501 (6)
Cl(1B)—C(2B)	1.735 (5)	C(2C)—C(3C)	1.381 (7)
Cl(2B)—C(4B)	1.734 (5)	C(3C)—C(4C)	1.371 (8)
Cl(3B)—C(6B)	1.727 (5)	C(4C)—C(5C)	1.383 (7)
O(1B)—C(7B)	1.233 (6)	C(5C)—C(6C)	1.372 (7)
O(2B)—C(7B)	1.250 (6)	O(2A^{i})···O(1A)	2.664 (3)
C(1B)—C(2B)	1.390 (6)	O(2A^{i})···H(1A)	1.69 (3)
C(1B)—C(6B)	1.392 (7)	Cl(2C)···Cl(2B^{ii})	3.437 (2)
C(1B)—C(7B)	1.503 (6)		
C(2A)—C(1A)—C(6A)	117.6 (4)	C(3B)—C(4B)—C(5B)	121.8 (5)
C(2A)—C(1A)—C(7A)	121.7 (4)	C(4B)—C(5B)—C(6B)	118.2 (5)
C(6A)—C(1A)—C(7A)	120.7 (4)	Cl(3B)—C(6B)—C(1B)	118.5 (3)
Cl(1A)—C(2A)—C(1A)	119.2 (3)	Cl(3B)—C(6B)—C(5B)	118.9 (4)
Cl(1A)—C(2A)—C(3A)	118.4 (3)	C(1B)—C(6B)—C(5B)	122.5 (4)
C(1A)—C(2A)—C(3A)	122.3 (4)	O(1B)—C(7B)—O(2B)	124.9 (5)
C(2A)—C(3A)—C(4A)	118.4 (4)	O(1B)—C(7B)—C(1B)	117.3 (4)
Cl(2A)—C(4A)—C(3A)	119.0 (4)	O(2B)—C(7B)—C(1B)	117.8 (4)
Cl(2A)—C(4A)—C(5A)	119.0 (3)	C(2C)—C(1C)—C(6C)	117.6 (4)
C(3A)—C(4A)—C(5A)	122.0 (4)	C(2C)—C(1C)—C(7C)	121.2 (4)
C(4A)—C(5A)—C(6A)	117.6 (4)	C(6C)—C(1C)—C(7C)	121.2 (4)
Cl(3A)—C(6A)—C(1A)	120.4 (3)	Cl(1C)—C(2C)—C(1C)	119.2 (4)
Cl(3A)—C(6A)—C(5A)	117.5 (3)	Cl(1C)—C(2C)—C(3C)	118.1 (4)
C(1A)—C(6A)—C(5A)	122.1 (4)	C(1C)—C(2C)—C(3C)	122.6 (4)
O(1A)—C(7A)—O(2A)	125.1 (4)	C(2C)—C(3C)—C(4C)	117.7 (4)
O(1A)—C(7A)—C(1A)	118.2 (4)	Cl(2C)—C(4C)—C(3C)	119.0 (4)
O(2A)—C(7A)—C(1A)	116.7 (4)	Cl(2C)—C(4C)—C(5C)	118.8 (4)
C(2B)—C(1B)—C(6B)	116.6 (4)	C(3C)—C(4C)—C(5C)	122.2 (5)
C(2B)—C(1B)—C(7B)	121.4 (4)	C(4C)—C(5C)—C(6C)	118.2 (5)
C(6B)—C(1B)—C(7B)	122.0 (4)	Cl(3C)—C(6C)—C(1C)	119.3 (3)
Cl(1B)—C(2B)—C(1B)	118.8 (4)	Cl(3C)—C(6C)—C(5C)	119.0 (4)
Cl(1B)—C(2B)—C(3B)	119.0 (4)	C(1C)—C(6C)—C(5C)	121.7 (4)
C(1B)—C(2B)—C(3B)	122.1 (4)	O(1C)—C(7C)—O(2C)	124.4 (5)
C(2B)—C(3B)—C(4B)	118.8 (4)	O(1C)—C(7C)—C(1C)	117.8 (4)
Cl(2B)—C(4B)—C(3B)	119.1 (4)	O(2C)—C(7C)—C(1C)	117.8 (4)
Cl(2B)—C(4B)—C(5B)	119.1 (4)		

Symmetry codes: (i) $1-x, 2-y, -z$; (ii) $\frac{3}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$.

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O(1A)—H(1A)···O(2A^{i})	0.98 (3)	1.69 (3)	2.664 (3)	172 (3)
O(1B)—H(1B)···O(2C)	0.98 (3)	1.71 (2)	2.661 (3)	166 (3)
O(1C)—H(1C)···O(2B)	0.98 (3)	1.72 (3)	2.693 (3)	176 (3)

Symmetry codes: (i) $1-x, 2-y, -z$.

All H atoms were found in difference Fourier maps and the non-carboxyl H atoms were allowed to refine as riding models; their average temperature factor was allowed to refine. The positional parameters of each of the carboxyl H atoms were refined, but these atoms were constrained to be 0.98 Å from their respective O atoms; the temperature factors of the carboxyl H atoms were held constant.

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXTL/PC*. Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1041). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(3a*R*,3b*S*,4a*R*,4b*R*,5a*R*,5b*S*)-4,4,8,8-Tetrabromo-2,2-dimethylperhydrodicyclopropa-[*e*,*g*][1,3]benzodioxole-3b-carbonitrile

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Abstract

The X-ray crystal structure of the title compound, $C_{12}H_{11}Br_4NO_2$, reveals that the two three-membered rings are in an *anti* relationship to one another.