Table 1. *Selected geometric parameters (Å, °)*

O1—C6	1.216 (3)	O3—C10	1.205 (5)
O2—C8	1.210 (3)	O4—C10	1.288 (5)
O3—C10—C9	124.0 (4)	O4—C10—C9	113.2 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O1^{i}	0.91 (6)	1.84 (6)	2.752 (3)	175 (1)
C12—H12*C*···O1^{ii}	0.96	2.58	3.509 (4)	163
C5—H5*A*···O2^{iii}	0.98	2.45	3.280 (3)	141
C12—H12*B*···O2^{iii}	0.96	2.70	3.621 (4)	160
C4—H4*A*···O3^{iv}	0.98	2.47	3.298 (4)	142

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

All non-carboxyl H atoms were found in electron-density difference maps but replaced in calculated positions and allowed to refine as riding models on their appropriate C atoms. Torsion angles for the methyl rotors were allowed to refine. Displacement parameters for the methine- and methylene-H atoms were refined as two groups and displacement parameters for the H atoms of each methyl group were fixed to be 150% of the isotropic displacement parameter of their associated C atom. The carboxyl-H atom was found in an electron-density difference map but was replaced in a calculated position and the O—H distance allowed to refine with its displacement parameter fixed to be 150% of the isotropic displacement parameter of its associated O atom.

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS* (Siemens, 1996). Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1994). Program(s) used to refine structure: *SHELXTL/PC*. Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1516). Services for accessing these data are described at the back of the journal.

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(±)-1-Indanone-2-acetic and -2-propionic acids: catemeric *versus* dimeric hydrogen bonding in homologous γ- and δ-keto acids

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Abstract

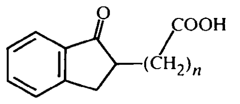
The crystal structure of (±)-2,3-dihydro-1-oxo-1*H*-indene-2-acetic acid ($C_{11}H_{10}O_3$) involves catemeric hydrogen bonds, which progress from the carboxyl H atom of one molecule to the ketone O atom of a neighbor screw-related in **b** [O···O = 2.709 (2) Å]. This yields centrosymmetrically related counterdirectional pairs of hydrogen-bonding chains following parallel helical axes. Four intermolecular C=O···H—C close contacts were found, involving both carbonyl groups. By contrast, the hydrogen bonding in (±)-3-(2,3-dihydro-1-oxo-1*H*-inden-2-yl)propionic acid ($C_{12}H_{12}O_3$) produces carboxyl dimers [O···O = 2.639 (2) Å], which also have a 2.62 Å C=O···H—C close contact involving the ketone carbonyl group and the *peri*-hydrogen on a centrosymmetrically related molecule. The difference in hydrogen-bonding behavior between the two molecules is discussed.

Comment

Our continuing interest in crystalline keto acids involves elucidation of factors controlling the one intra- and four intermolecular modes that have thus far been found to characterize their hydrogen bonding. Most commonly, carboxyl groups are paired by mutual hydrogen bonding, without ketone participation, to form the common acid-to-acid dimer motif (Thompson, Lalancette & Brunskill, 1998). Less frequently a chain (catemer) is produced by repetition of an intermolecular carboxyl-to-ketone hydrogen bond (Zewge *et al.*, 1998). Relatively rare arrangements involve internal hydrogen bonds (Thompson *et al.*, 1996), acid-to-ketone dimers (Kosela *et al.*, 1995) and carboxyl catemers (Lalancette *et al.*, 1998). Several cases also exist of hydrates with more complex hydrogen-bonding patterns (Lalancette *et al.*, 1997; 1998).

We have recently discussed several factors that contribute to the choice of hydrogen-bonding mode, including the unavailability of an inversion center for enantiomerically pure compounds (Brunskill *et al.*, 1999). Another such general factor, which appears to favor simple acid–dimer pairing over the formation of catemers, is flexibility, which allows the molecule to find a packable conformation for the dimer. Overall flexibility increases with the length of $(CH_2)_n$ chains holding the acid or ketone functions, and we know of only one racemic catemeric keto acid having such a chain longer than $n = 2$ (Abell *et al.*, 1991). Given a system that tends to produce catemers, a question of interest is how long a polymethylene chain attaching either function will be tolerated, before the bias toward catemers is overcome in favor of dimerization.

We have established that the 1-indanone system has a particular propensity to produce catemers in the solid state (Brunskill *et al.*, 1997; Lalancette *et al.*, 1997; Thompson, Brunskill & Lalancette, 1998). In a wide variety of other keto acids, the observed tendency to form catemers seems generally to be greatest for γ-acids. Therefore, 1-indanones with acid chain-lengths in the range of $n = 1$ and 2 appeared to constitute a test system for the above question, and we now report the crystal structures for the title compounds, homologous γ-, (I), and δ-, (II), keto acids which are racemic 2-substituted 1-indanones.



(I) $n = 1$
(II) $n = 2$

Fig. 1 shows the asymmetric unit for (I) with its numbering scheme. The molecule is skeletally rigid, except for rotation about the C2—C8 and C8—C9 bonds. The C3a—C3—C2—C8 torsion angle is 140.6 (2)°, and the substituents attached to C2 and C8 are staggered [C3—C2—C8—C9 torsion angle = −69.4 (2)°]. The carboxyl is rotated, with its C=O lying over the cyclopentanone ring [C2—C8—C9—O2 torsion angle = 4.9 (3)°]. This conformation gives the carboxyl an 80.8 (1)° dihedral angle relative to the aromatic ring. A slight deviation of C2 from the general bicyclic plane produces a shallow dihedral angle of 13.6 (2)° between that plane (omitting C2) and C1—C2—C3 [*cf.* 7.2 (3) and 13.0 (1)° for (±)-1-indanone-3-carboxylic (Lalancette *et al.*, 1997) and -3-acetic acids (Thompson, Brunskill & Lalancette, 1998), respectively]. As is characteristic of catemers, the carboxyl group is fully ordered, with bond lengths of 1.199 (2) and 1.328 (2) Å for C=O and C—O, respectively.

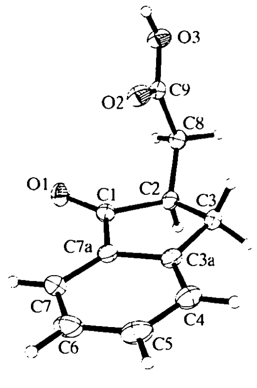


Fig. 1. The asymmetric unit of (I), with its atom-numbering scheme. Ellipsoids are drawn at the 20% probability level, and H atoms are shown as spheres of an arbitrary radius.

Fig. 2 shows the packing arrangement for (I). Carboxyl-to-ketone catemers [O···O = 2.709 (2) Å and O—H···O = 175°] are formed by hydrogen bonds that progress among molecules screw-related in **b** [infinite chains with base vector (0,1,0)]. Centrosymmetrically related single-strand helices proceed counter-directionally through the cell. This hydrogen-bonding pattern conforms to the commonest of the sub-types of acid-to-ketone catemer described above, categorized by the symmetry between adjacent chain members. In order of decreasing prevalence, these symmetry relations are: screw, translation (collectively, 'homochiral'), and glide ('heterochiral'). Four intermolecular C=O···H—C close contacts were found, involving both the ketone (2.64 Å to H2*A*) and the acid (2.52 to H2*A*, 2.68 to H3*A* and 2.56 Å to H6*A*). Such contacts probably represent polar attractions contributing materially to the packing forces (Jönsson, 1972; Leiserowitz, 1976; Berkovitch-Yellin & Leiserowitz, 1982).

Fig. 3 shows the asymmetric unit for (II) with its numbering scheme. The carboxyethyl chain adopts the staggered and flattened conformation shown [torsion angles: C3—C2—C8—C9 = 60.0 (3) and C2—C8—C9—C10 = 177.9 (2)°], and the carboxyl unit is rotated,

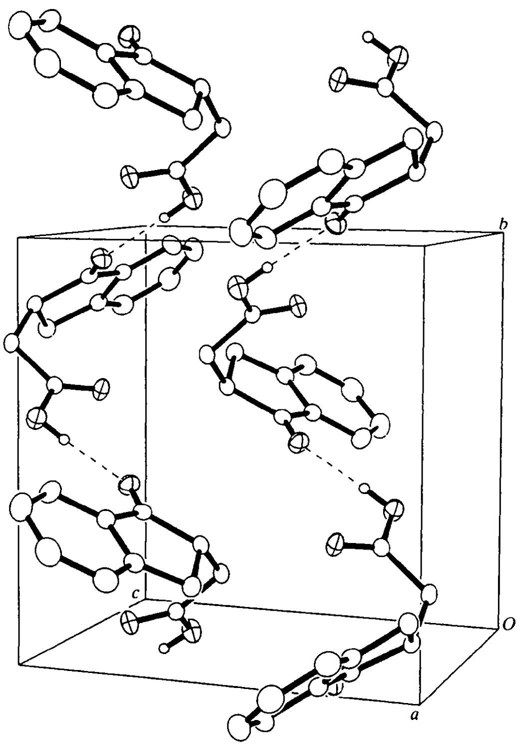


Fig. 2. A partial packing diagram for (I), with extracellular molecules, showing one pair of parallel counterdirectional centrosymmetrically related single-strand hydrogen-bonding helices. For clarity, all carbon-bound H atoms have been omitted. Ellipsoids are drawn at the 20% probability level.

with its carbonyl in the direction of the ketone [C8—C9—C10—O2 = −9.9 (3)°]. These rotations cause the carboxyl and its chain to be more nearly parallel to the ring sytem than is seen in (I), and the carboxyl makes a dihedral angle of 34.7 (1)° with the aromatic ring. The dihedral angle describing the slight deviation of C2 from the general bicyclic plane is 12.8 (2)°, compared with 13.6 (2)° for (I). The carboxyl group displays negligible disorder, with bond lengths of 1.211 (2) for C═O and 1.310 (2) Å for C—O, and angles of 123.6 (2) and 112.9 (2)°.

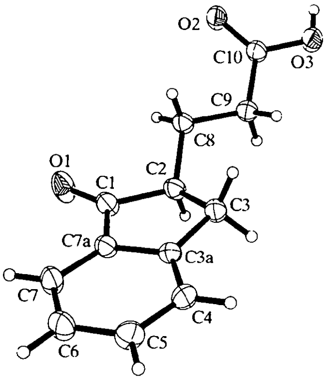


Fig. 3. The asymmetric unit of (II), with its atom-numbering scheme. Ellipsoids are drawn at the 20% probability level, and H atoms are shown as spheres of an arbitrary radius.

Fig. 4 illustrates the packing of (II) with acid dimers [O···O = 2.639 (2) Å and O—H···O = 172°] centered on the *bc* face and on the corners of the chosen cell. Intermolecular C═O···H—C close contacts of 2.62 Å were found, involving the ketone and the *peri*-hydrogen, H7, on centrosymmetrically related (non-hydrogen-bonded) molecules. These contacts thus cause a chaining together of acid–dimer pairs, forming an extended ribbon-like network.

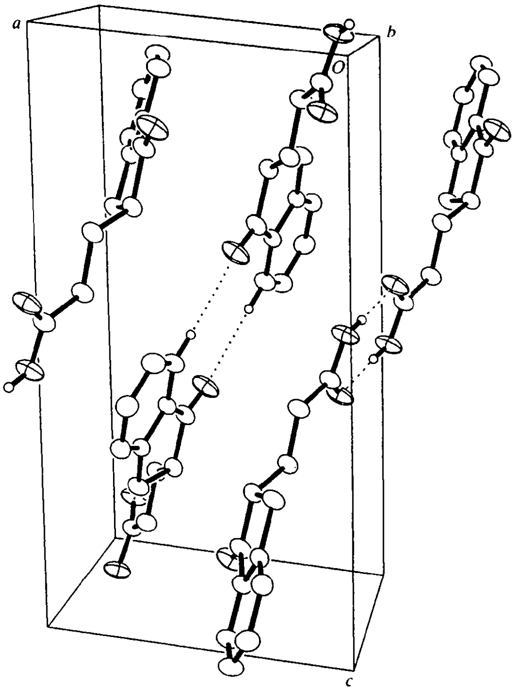


Fig. 4. A partial packing diagram for (II), with extracellular molecules, illustrating the acid dimer centered on the *bc* face and molecules linked by reciprocal close contacts across the center of the cell. For clarity, all carbon-bound H atoms have been omitted except those involved in the reciprocal close contact. Ellipsoids are drawn at the 20% probability level.

In considering the factors that cause the observed change in hydrogen-bonding mode, the geometries of the two different types of hydrogen bond are of interest. One important factor affecting the strength (and length) of a hydrogen bond is the angle of approach of the acid proton to the carbonyl acceptor. This can be characterized using the H···O═C angle and H···O═C—*X* torsion angle, where *X* = C for a ketone acceptor and *X* = O for an acid acceptor. These angles describe the approach of the acid proton to the oxygen 'lone-pair lobes', which theoretically lie at 120° to the C═O bond and in the carbonyl plane. An 'ideal' hydrogen bond would have values of H···O═C = 120° and H···O═C—*X* = 0°. For the catemer, (I), these angles are 132 and −3.0°, respectively, while for the dimer, (II), they are 122 and −0.2°, respectively. The slightly longer hydrogen-bond distance found for the catemer, (I) [O···O = 2.709 (2) Å], *versus* the dimer, (II) [O···O = 2.639 (2) Å], is consistent with the somewhat poorer hydrogen-bond geometry for the catemer. However, the apparently weaker hydrogen bond in the catemer system seems to be compensated for by more numerous attractive close contacts and a higher packing efficiency. For (I), the density is 1.360 Mg m^{-3}, with the space filled by the molecule calculated to be 69.3%. For (II), the density is lower, at 1.297 Mg m^{-3}, and the space filled is correspondingly reduced to 67.4%.

Our X-ray structures of five varied 1-indanone carboxylic acids, including the present two (Brunskill *et al.*, 1997; Lalancette *et al.*, 1997; Thompson, Brunskill & Lalancette, 1998), have found only one which is not catemerically hydrogen bonded. This contrasts markedly with four closely analogous cases containing the 1-tetralone skeleton (Thompson, Lalancette & Brunskill, 1998, and unpublished work), which are all carboxyl dimers. It appears, therefore, that the 1-indanone system bears some inherent tendency to produce catemers, most probably due to the rigidity of the five-membered ring (enhanced by the conjugation of the ketone with the aromatic ring). Since the only difference between (I) and (II) lies in the added member of the carboxyl chain, we attribute the change toward the dimeric hydrogen-bonding mode in (II) to the increased flexibility associated with this chain lengthening. It is to be noted in (I) and (II) that the C3—C2—C8—C9 torsion angles differ by 129.4 (2)°; in (I), the C8—C9 bond of the carboxyl-bearing chain projects upward, approximately orthogonal to the ring plane, whereas in (II), it extends in a direction generally parallel to the ring-plane, creating a relatively flat stackable unit. Flattening of the extended chain in (II) is further enhanced by the conformation of the carboxyl group.

We believe that the difference in conformational flexibility in (I) *versus* (II) exemplifies a significant general factor affecting the choice of hydrogen-bonding mode in keto carboxylic acids. However, the evidence is far from perfect (*cf.* Lalancette *et al.* 1991; Zewge *et al.*, 1998), and we are pursuing other examples which may provide data on this point.

The solid-state (KBr) IR spectrum of (I) has C═O absorptions at 1740 and 1671/1667 cm^{-1}. These positions conform to the shifts seen typically in catemers, due, respectively, to removal of hydrogen bonding from carboxyl C═O and addition of hydrogen bonding to the ketone (*cf.* 1728 and 1673 cm^{-1} for 1-indanone-3-acetic acid, and 1736 and 1679 cm^{-1} for 1-indanone-3-carboxylic acid). In $CHCl_3$ solution, the absorptions for (I) coalesce to a single broad peak centered at 1712 cm^{-1}, consistent with a dimerically hydrogen-bonded carboxyl group (*cf.* 1712 and 1715 cm^{-1} for 1-indanone-3-acetic and -3-carboxylic acids, respectively). In (II), the C═O regions are essentially identical for the KBr and $CHCl_3$ spectra, with a single peak at 1710–1711 cm^{-1}, consistent with a dimerically hydrogen-bonded carboxyl group.

Experimental

Both (I) and (II) were synthesized by saponification of the distilled product obtained from alkylation of the pyrrolidine enamine (Stork *et al.*, 1963) of 1-indanone (Thompson & Huegi, 1976), using ethyl bromoacetate and ethyl acrylate, respectively. For (I), crystals of X-ray quality (m.p. 423 K) were obtained from EtOAc and from *tert*-butyl methyl ether. For (II), crystals (m.p. 381 K) were obtained from Et_2O/CH_2Cl_2.

Compound (I)

Crystal data

$C_{11}H_{10}O_3$
$M_r = 190.19$
Monoclinic
$P2_1/c$
$a = 8.945\,(1)$ Å
$b = 10.318\,(1)$ Å
$c = 10.127\,(1)$ Å
$\beta = 96.40\,(1)°$
$V = 928.8\,(2)$ Å^3
$Z = 4$
$D_x = 1.360$ Mg m^{-3}
$D_m = 1.36\,(1)$ Mg m^{-3}
D_m measured by flotation in CCl_4/*p*-xylene

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 31 reflections
$\theta = 2.81–13.60°$
$\mu = 0.099$ mm^{-1}
$T = 293\,(2)$ K
Tabular
0.68 × 0.40 × 0.12 mm
Colorless

Data collection

Siemens *P*4 diffractometer
$2\theta/\theta$ scans
Absorption correction: face-indexed numerical (Sheldrick, 1997)
$T_{\min} = 0.951$, $T_{\max} = 0.989$
2215 measured reflections
1643 independent reflections
1194 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.039$
$\theta_{\max} = 25°$
$h = -10 \rightarrow 10$
$k = -1 \rightarrow 12$
$l = 0 \rightarrow 12$
3 standard reflections every 97 reflections intensity variation: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.108$
$S = 1.03$
1643 reflections
137 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0387P)^2 + 0.2532P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.150$ e Å^{-3}
$\Delta\rho_{\min} = -0.137$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °) for (I)*

O1—C1	1.225 (2)	O3—C9	1.328 (2)
O2—C9	1.199 (2)		
O2—C9—C8	123.9 (2)	O3—C9—C8	113.3 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1^{i}	0.82	1.89	2.709 (2)	175
C2—H2*A*···O1^{ii}	0.98	2.64	3.421 (2)	137
C2—H2*A*···O2^{iii}	0.98	2.52	3.156 (2)	122
C3—H3*A*···O2^{iii}	0.97	2.68	3.311 (3)	123
C6—H6*A*···O2^{iv}	0.93	2.56	3.245 (3)	131

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

Compound (II)

Crystal data

$C_{12}H_{12}O_3$
$M_r = 204.22$
Monoclinic
$P2_1/c$
$a = 8.093$ (1) Å
$b = 8.326$ (1) Å
$c = 15.614$ (2) Å
$\beta = 96.20$ (1)°
$V = 1046.0$ (2) Å^3
$Z = 4$
$D_x = 1.297$ Mg m^{-3}
$D_m = 1.29$ (1) Mg m^{-3}
D_m measured by flotation in CCl_4/*p*-xylene

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 45 reflections
$\theta = 4.64$–17.30°
$\mu = 0.093$ mm^{-1}
$T = 293$ (2) K
Tabular
0.70 × 0.62 × 0.40 mm
Pale yellow

Data collection

Siemens *P*4 diffractometer
$2\theta/\theta$ scans
Absorption correction: face-indexed numerical (Sheldrick, 1997)
$T_{\min} = 0.947$, $T_{\max} = 0.968$
2862 measured reflections
2056 independent reflections
1452 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.023$
$\theta_{\max} = 26°$
$h = -9 \rightarrow 9$
$k = -1 \rightarrow 10$
$l = 0 \rightarrow 19$
3 standard reflections every 97 reflections intensity variation: ±1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.134$
$S = 1.06$
2055 reflections
148 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0557P)^2 + 0.2204P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.165$ e Å^{-3}
$\Delta\rho_{\min} = -0.115$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 3. *Selected geometric parameters (Å, °) for (II)*

O1—C1	1.215 (2)	O3—C10	1.310 (2)
O2—C10	1.211 (2)		
O2—C10—C9	123.6 (2)	O3—C10—C9	112.9 (2)

Table 4. *Hydrogen-bonding geometry (Å, °) for (II)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2^{i}	0.82	1.83	2.639 (2)	172
C7—H7*A*···O1^{ii}	0.93	2.62	3.381 (3)	140

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

All H atoms for both (I) and (II) were found in electron-density difference maps, but were replaced in calculated positions and allowed to refine as riding models on their appropriate C atoms, with their displacement factors free to refine.

For both compounds, data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structures: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXL*97; software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1525). Services for accessing these data are described at the back of the journal.

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