

O(2)...O(1)' = 2.753 (3) Å, O(2)...O(1)' = 2.794 (3) Å and N(1)...O(2)' = 2.665 (3) Å; symmetry codes: $x, y, z; x, -\frac{1}{2}-y, -\frac{1}{2}+z; -x, \frac{1}{2}+y, \frac{1}{2}-z$.

The packing of the tautomer (II) is characterized by chains of hydrogen-bonded molecules. These chains are built up by only one type of hydrogen bond: O(1)—H(1)...N(3)' [O(1)...N(3)' = 2.586 (2) Å; symmetry code: $1\frac{1}{2}-x, -\frac{1}{2}+y, 1\frac{1}{2}-z$] and the chains are themselves held together by close contacts of antiparallel, centrosymmetrically related triazole rings, suggesting some charge-transfer interactions. The contact distances of the relevant atoms lie within the range 3.4–3.7 Å (symmetry codes: $1\frac{1}{2}-x, \frac{1}{2}-y, 1-z; 1-x, -y, 1-z$).

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* See deposition footnote.

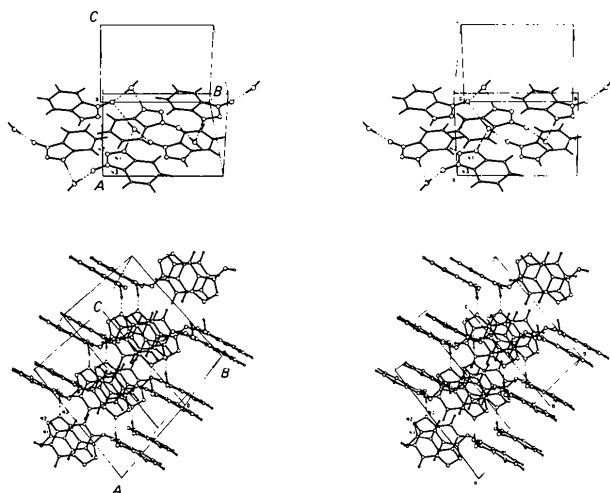


Fig. 4. (a) Crystal packing of (I). (b) Crystal packing of (II). Hydrogen bonds are illustrated with dashed lines.

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2-Benzoylcyclohexanecarboxylic Acid, C₁₄H₁₆O₃

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Abstract. $M_r = 232.28$, monoclinic, $C2/c$, $a = 17.103$ (6), $b = 6.758$ (2), $c = 22.149$ (7) Å, $\beta = 101.20$ (3)°, $V = 2511$ (2) Å³, $D_x = 1.22$ g cm⁻³, $Z = 8$, Mo $K\alpha$ ($\lambda = 0.71069$ Å), $\mu = 1.00$ cm⁻¹, $F(000) =$

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Introduction. Metal hydride reduction of the 2-benzoyl-cyclohexanecarboxylic acids (Fieser & Novello, 1942) provides the corresponding hydroxy acids with high selectivity for obedience of the Cram rule. Platinum oxide-catalyzed hydrogenation, however, favors formation of the anti-Cram product but with less selectivity. Intramolecular association of functional groups, carboxylate anion and ketone carbonyl carbon in the former case and hydrogen bonding of carboxyl group to ketone carbonyl oxygen in the latter case (Pourahmady & Eisenbraun, 1982; Pourahmady, 1981) is assumed to control the product stereochemistry. We have determined the solid-state structure of the *cis*-keto acid to investigate the possibility of intramolecular interaction of functional groups in the solid state.

Experimental. A crystal of $C_{14}H_{16}O_3$ ($0.1 \times 0.3 \times 0.25$ mm), recrystallized from acetic acid, was mounted on a Syntex P3 diffractometer; fifteen reflections ($15^\circ < 2\theta < 30^\circ$) used for determination of lattice parameters during normal alignment procedures; systematic absences: hkl $h + k = 2n + 1$, $h0l$ $l = 2n + 1$ ($h = 2n + 1$), $0k0$ ($k = 2n + 1$); 3293 independent reflections measured, 1285 observed [$I > 3\sigma(I)$], $0^\circ < 2\theta < 116^\circ$; h -17 to +19, k 0 to 8, l 0 to 20; three standards remeasured every 97 reflections, variation <5%; data corrected for Lp effects, structure solved using *MULTAN80* (Main *et al.*, 1980), refined by full-matrix least-squares calculations [function

minimized $\sum(|F_o| - |F_c|)^2$]; H atoms located from difference Fourier synthesis; final refinement included scale factor, positional parameters for all atoms, anisotropic thermal parameters for nonhydrogen atoms, isotropic thermal parameters for H atoms; unit weights used throughout; normal scattering factors (Cromer & Mann, 1968), XRAY78 (Stewart, 1978); $R = 4.0\%$, final difference Fourier map featureless, ratio of maximum least-squares shift to error in final refinement = 0.28.

Discussion. Table 1 shows atomic coordinates, Table 2 bond distances and angles, and dihedral angles.* Fig. 1 is a projection of the molecule.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38539 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates with *e.s.d.*'s in parentheses and isotropic thermal parameters

U_{eq} (non-hydrogen atoms) is $\frac{1}{3}$ the trace of the diagonalized anisotropic matrix. U_{eq} and U_{iso} are in units of $\text{\AA}^2 \times 10^3$.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}/U_{iso}
C(1)	0.3584 (1)	-0.0012 (4)	0.2372 (1)	41
C(2)	0.3096 (2)	0.1309 (5)	0.2604 (1)	55
C(3)	0.3013 (2)	0.1175 (6)	0.3210 (2)	70
C(4)	0.3414 (2)	-0.0238 (6)	0.3589 (2)	69
C(5)	0.3890 (2)	-0.1558 (6)	0.3365 (2)	68
C(6)	0.3970 (2)	-0.1467 (5)	0.2760 (1)	51
C(7)	0.3704 (1)	0.0029 (4)	0.1720 (1)	41
C(8)	0.3469 (2)	0.1867 (4)	0.1333 (1)	49
C(9)	0.3952 (2)	0.3688 (5)	0.1603 (2)	67
C(10)	0.4802 (2)	0.3589 (6)	0.1511 (2)	74
C(11)	0.4837 (3)	0.3433 (6)	0.0832 (2)	81
C(12)	0.4396 (2)	0.1600 (6)	0.0552 (2)	69
C(13)	0.3542 (2)	0.1547 (5)	0.0656 (1)	57
C(14)	0.3098 (2)	-0.0260 (5)	0.0377 (1)	59
O(1)	0.3982 (1)	-0.1400 (3)	0.1507 (1)	61
O(2)	0.2398 (1)	-0.0509 (4)	0.0530 (1)	76
O(3)	0.3351 (1)	-0.1357 (4)	0.0018 (1)	81
H(1)	0.215 (2)	-0.171 (7)	0.031 (2)	149 (18)
H(2)	0.282 (2)	0.230 (4)	0.235 (1)	61 (9)
H(3)	0.271 (2)	0.201 (5)	0.338 (1)	76 (11)
H(4)	0.335 (2)	-0.029 (4)	0.402 (1)	75 (10)
H(5)	0.413 (2)	-0.253 (5)	0.360 (1)	82 (12)
H(6)	0.430 (1)	-0.236 (4)	0.259 (1)	52 (8)
H(8)	0.289 (2)	0.205 (4)	0.135 (1)	52 (9)
H(13)	0.325 (2)	0.265 (4)	0.044 (1)	59 (8)
H(91)	0.394 (2)	0.381 (5)	0.207 (2)	97 (12)
H(92)	0.369 (2)	0.482 (5)	0.139 (1)	74 (10)
H(101)	0.508 (2)	0.239 (5)	0.171 (1)	80 (11)
H(102)	0.506 (2)	0.477 (5)	0.167 (1)	89 (10)
H(111)	0.537 (2)	0.340 (5)	0.076 (1)	83 (11)
H(112)	0.457 (2)	0.467 (5)	0.060 (2)	98 (12)
H(121)	0.436 (2)	0.160 (5)	0.012 (2)	89 (12)
H(122)	0.467 (2)	0.040 (4)	0.074 (1)	66 (10)

Table 2. Bond distances (\AA), bond angles ($^\circ$) and dihedral angles ($^\circ$)

C(1)-C(2)	1.387 (4)	C(1)-C(2)-C(3)	120.1 (3)
C(1)-C(6)	1.386 (4)	C(2)-C(3)-C(4)	120.6 (4)
C(2)-C(3)	1.380 (5)	C(3)-C(4)-C(5)	119.8 (4)
C(3)-C(4)	1.365 (6)	C(4)-C(5)-C(6)	120.3 (4)
C(4)-C(5)	1.364 (6)	C(5)-C(6)-C(1)	120.7 (3)
C(5)-C(6)	1.374 (5)	C(2)-C(1)-C(6)	118.4 (2)
C(1)-C(7)	1.498 (3)	C(6)-C(1)-C(7)	118.2 (2)
C(7)-C(8)	1.518 (4)	C(2)-C(1)-C(7)	123.4 (2)
C(7)-O(1)	1.212 (3)	C(1)-C(7)-C(8)	119.2 (2)
C(8)-C(9)	1.537 (4)	C(1)-C(7)-O(1)	119.9 (2)
C(8)-C(13)	1.544 (3)	C(8)-C(7)-O(1)	120.9 (2)
C(9)-C(10)	1.508 (5)	C(7)-C(8)-C(9)	111.8 (2)
C(10)-C(11)	1.520 (6)	C(7)-C(8)-C(13)	111.8 (2)
C(11)-C(12)	1.519 (6)	C(9)-C(8)-C(13)	110.6 (3)
C(12)-C(13)	1.523 (5)	C(8)-C(9)-C(10)	111.8 (3)
C(13)-C(14)	1.506 (4)	C(9)-C(10)-C(11)	111.2 (3)
C(14)-O(2)	1.317 (4)	C(10)-C(11)-C(12)	110.4 (4)
C(14)-O(3)	1.226 (4)	C(11)-C(12)-C(13)	111.9 (3)
O(2)-H(1)	1.00 (4)	C(12)-C(13)-C(8)	113.9 (2)
H(1)-O(3')	1.65 (4)	C(12)-C(13)-C(14)	112.3 (3)
		C(8)-C(13)-C(14)	112.5 (2)
		C(13)-C(14)-O(2)	114.2 (3)
		C(13)-C(14)-O(3)	122.9 (3)
		O(2)-C(14)-O(3)	122.9 (3)
		C(14)-O(2)-H(1)	107 (2)
		O(2)-H(1)-O(3')	173 (4)
		O(1)-C(7)-C(8)-C(13)	6.4 (3)
		O(1)-C(7)-C(8)-C(9)	-118.2 (3)
		O(1)-C(7)-C(1)-C(2)	-163.1 (2)
		O(1)-C(7)-C(1)-C(6)	15.7 (3)
		H(1)-O(2)-C(14)-O(3)	1.0 (20)
		H(1)-O(2)-C(14)-C(13)	178.1 (26)
		O(3)-C(14)-C(13)-C(12)	-12.1 (4)
		O(3)-C(14)-C(13)-C(8)	-142.1 (3)
		O(2)-C(14)-C(13)-C(8)	40.8 (3)
		O(2)-C(14)-C(13)-C(12)	170.8 (2)
		C(14)-C(13)-C(8)-C(7)	53.0 (3)

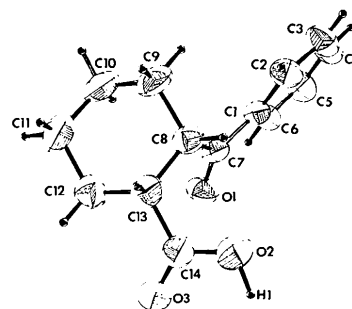


Fig. 1. Projection of the $C_{14}H_{16}O_3$ molecule.

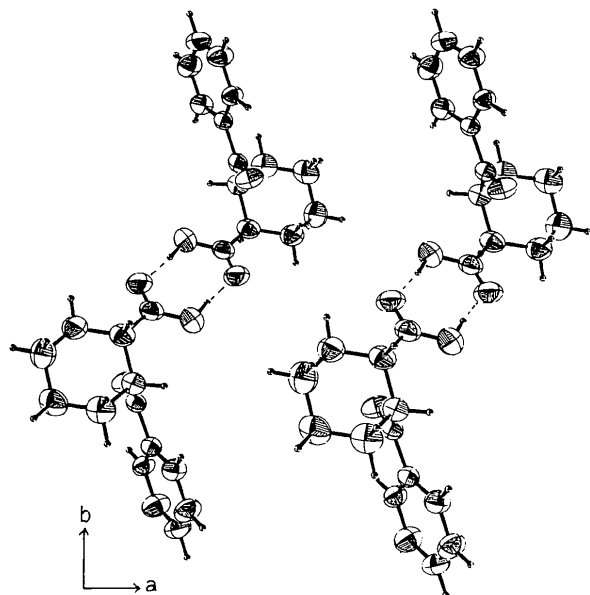


Fig. 2. Projection of the unit cell (*ab* plane) showing molecules in symmetry modifications x, y, z ; $\frac{1}{2}-x, -\frac{1}{2}-y, -z$; $-x, -y, -z$; and $-\frac{1}{2}+x, \frac{1}{2}+y, z$. Dashed lines are intermolecular hydrogen bonds.

The eight molecules of 2-benzoylcyclohexanecarboxylic acid exist in pairs (Fig. 2) bonded together with two intermolecular hydrogen bonds involving the

carboxylic groups [O(2)—H(1), 1.00 (4); H(1)···O(3'), 1.65 (4) Å, \angle O(2)—H(1)···O(3'), 173 (4)°]. The benzoyl oxygen atom, O(1), is not involved in hydrogen bonding and does not show the intramolecular hydrogen bonding postulated [O(2)—H(1)···O(1)] for the molecule in solution. [All O(1) intermolecular distances exceed 3.0 Å.] The cyclohexane ring exists in the chair form with the benzoyl group in the axial position and the carboxylic substituent in the equatorial position as predicted by conformational theory. All angles and distances are normal. There are no other significant intermolecular contact distances.

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Structure of 5,6,7,8-Tetrahydro-4*H*-3,1-benzothiazine-2(1*H*)-spirocyclohexane-4-thione, C₁₃H₁₉NS₂

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Abstract. $M_r = 253.42$, orthorhombic, $Pcab$, $a = 11.059$ (1), $b = 20.348$ (1), $c = 11.578$ (1) Å, $V = 2605.3$ (3) Å³, $Z = 8$, $D_m = 1.31$, $D_x = 1.29$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.367$ mm⁻¹, $F(000) = 1088$, $T = 293$ K. Final $R = 0.034$ for 1734 observed reflections. The conformational parameters of the title compound are compared with those of other compounds. The deformation energies related to real chair form and real half-chair form in cyclohexane and

cyclohexene rings are 0.14 (1) and 1.45 (1) kJ mol⁻¹ respectively. The amount of double-bond character in the exocyclic C=S bond is about 75%.

Introduction. A great deal of research in the field of coordination chemistry has centred on complexes with ligands in which sulphur is the donor atom, or metallic chelates with ligands containing nitrogen and sulphur donors. Metallic complexes involving ligands with a