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Two Structures Involved in the Separation of the Enantiomeric Pair of 2,2′-Spirobi[indan]-1,1′-dione

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Abstract. (1*R*,2′*S*)-1′-Oxo-2,2′-spirobi[indan]-1-yl (1*S*)-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate (1), $C_{27}H_{26}O_5$, $M_r = 430.51$, monoclinic, $P2_1$, $a = 18.805$ (2), $b = 6.318$ (1), $c = 9.988$ (1) Å, $\beta = 112.41$ (1)°, $V = 1097.1$ (1) Å^3, $Z = 2$, $D_x = 1.303$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.83$ cm^{-1}, $F(000) = 456$, room temperature, $R = 0.040$, 2754 independent reflections [$I > 3\sigma(I)$], no extinction correction. (1*S*,2′*R*)-1′-Oxo-2,2′-spirobi[indan]-1-yl (1*S*)-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate (2), $C_{27}H_{26}O_5$, $M_r = 430.51$, monoclinic, $P2_1$, $a = 18.551$ (2), $b = 6.495$ (1), $c = 9.448$ (1) Å, $\beta = 96.521$ (9)°, $V = 1131.0$ (2) Å^3, $Z = 2$, $D_x = 1.264$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.81$ cm^{-1}, $F(000) = 456$, room temperature, $R = 0.062$, 1408 independent reflections [$I > 3\sigma(I)$], no extinction correction. The enantiomeric pair of (±)-2,2′-spirobi[indan]-1,1′-dione can be separated by repeated recrystallization after conversion into the diastereomeric pair (1*R*,2′*S*)- (1), and (1*S*,2′*R*)-1′-oxo-2,2′-spirobi[indan]-1-yl (−)-ω-camphanoate, (2). The configurations of (1) and (2) have been determined by X-ray crystallographic analyses.

Introduction. Separation of an enantiomeric pair into its two optically active pure species has always been difficult. The preparation of (+)-2,2′-spirobi[indan]-1,1′-dione has been reported (Dynesen, 1975). The essential step involves the resolution of *trans*-(±)-1′-hydroxy-2,2′-spirobi[indan]-1-one by means of the camphanic ester. The exact configurations of the two camphanic esters found from an X-ray crystallographic analysis are reported here. Some comments on the slightly different physical properties of the two camphanic ester derivatives are given. Furthermore, some of the results from an ^{1}H and ^{13}C NMR study are discussed in relation to the crystal structure.

Experimental. A mixture of (±)-2,2′-spirobi[indan]-1,1′-dione was prepared according to Dynesen (1972). A stereoselective reduction of one of the carbonyl groups in the diketone was performed with lithium tri-*tert*-butoxylaluminium hydride. The resulting alcohol group in the racemic *trans*-hydroxy ketone was transformed into an ester by acylation with (−)-ω-camphanic acid chloride conserving the geometry of the reduced carbon. The two diastereomeric esters formed, (1) and (2), were separated by fractional crystallization. (1) had lower solubility in acetone than (2) and was isolated as nice pure crystals in the first fractions. (2), with a lower melting point than (1), was more difficult to isolate, and had to be recrystallized several times to obtain a single crystal useful for X-ray analysis.

Equidimensional single-crystal fragments of maximum dimensions less than 0.5 mm were used for the crystallographic examinations. The space groups of both crystals were determined from precession photographs to be monoclinic with only systematic absences $0k0$ for k odd indicating $P2_1$. The lattice constants of both compounds were refined from approximately 25 centred reflections in the range $20 < 2\theta < 40°$. Intensities were measured using the $\omega/2\theta$ scan mode on a Huber diffractometer using Nb-filtered Mo $K\alpha$ radiation. Three standard reflections were measured every 100 reflections. For (1), for $2\theta \le 60°$ ($0 \le h \le 26$, $0 \le k \le 8$, $-14 \le l \le 12$), 6042 reflections were measured, of which 2942 reflections with $I > 3\sigma(I)$ were used for structure determination and refinements. It was observed that intensities drop more quickly with $\sin\theta/\lambda$ for (2) than for (1), and so for (2) data were collected for $2\theta \le 50°$ ($-24 \le h \le 24$, $0 \le k \le 8$, $0 \le l \le 12$), giving 2979 unique reflections, of which 1408 reflections had $I > 3\sigma(I)$. The intensities were corrected for Lorentz and polarization effects. $\mu(1) = 0.83$ cm^{-1} and $\mu(2) = 0.81$ cm^{-1}, thus no absorption corrections were applied. Positions of all C and O atoms in the structures were found by direct methods (Sheldrick, 1990). H-atom positions were found from difference Fourier maps. The structures were refined by full-matrix least-squares techniques using anisotropic thermal parameters for the non-H atoms and iso-

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tropic thermal parameters for the H atoms. For (1), $R = 0.040$ and $wR = 0.053$, $w = 1/\sigma^2(F)$, $\sigma(F) = [\sigma(F^2) + 1.03F^2]^{1/2} - |F|$, $S = 1.16$, $(\Delta/\sigma)_{\max} = 0.03$, $|\Delta\rho|_{\max} = 0.17$, no extinction correction. For (2), $R = 0.062$ and $wR = 0.074$, $w = 1/\sigma^2(F)$, $\sigma(F) = [\sigma(F^2) + 1.05F^2]^{1/2} - |F|$, $S = 1.53$, $(\Delta/\sigma)_{\max} = 0.09$, $|\Delta\rho|_{\max} = 0.31$, no extinction correction.

All calculations were performed on a VAX 6210 computer with the following programs: *INTEG*, based on the Nelmes (1975) algorithm for integration of intensities; *DATAP, DSORT* and *LINEX* (Coppens, 1974), for further data processing and least-squares refinement; *ORFFE* (Busing, Martin & Levy, 1964) for geometry; and *ORTEP* (Johnson, 1976) for molecular illustration. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final fractional coordinates and equivalent isotropic temperature factor coefficients are given in Table 1.* Geometric details of the structures are given in Table 2.

Table 1. *Fractional coordinates and equivalent isotropic temperature factors* (Å^2) *for* (1) *and* (2) *with e.s.d.'s in parentheses*

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

	x	y	z	$U_{\rm eq}$
Diastereoisomer (1)				
C1	0.0842 (1)	0.8266	−0.1968 (2)	0.035 (1)
C2	0.1292 (1)	1.0160 (6)	−0.2199 (2)	0.035 (1)
C3	0.1508 (2)	1.1503 (6)	−0.0808 (3)	0.044 (1)
C4	0.1283 (1)	1.0161 (6)	0.0217 (2)	0.037 (1)
C5	0.1391 (2)	1.0570 (7)	0.1646 (3)	0.051 (1)
C6	0.1115 (2)	0.9129 (8)	0.2374 (3)	0.060 (2)
C7	0.0748 (2)	0.7295 (7)	0.1711 (3)	0.055 (2)
C8	0.0631 (1)	0.6867 (6)	0.0288 (3)	0.044 (1)
C9	0.0900 (1)	0.8330 (6)	−0.0446 (2)	0.034 (1)
O1	0.0498 (1)	0.6981 (6)	−0.2885 (2)	0.053 (1)
C1′	0.2005 (1)	0.9415 (6)	−0.2500 (2)	0.034 (1)
C3′	0.0839 (1)	1.1444 (7)	−0.3591 (3)	0.046 (1)
C4′	0.1468 (1)	1.2379 (6)	−0.3996 (2)	0.038 (1)
C5′	0.1459 (2)	1.4117 (7)	−0.4837 (3)	0.049 (1)
C6′	0.2128 (2)	1.4687 (7)	−0.5022 (3)	0.058 (2)
C7′	0.2797 (2)	1.3542 (8)	−0.4377 (3)	0.061 (2)
C8′	0.2807 (2)	1.1752 (7)	−0.3551 (3)	0.052 (1)
C9′	0.2136 (1)	1.1209 (6)	−0.3366 (2)	0.036 (1)
O2	0.2708 (1)	0.9112 (5)	−0.1237 (2)	0.040 (1)
C	0.2798 (1)	0.7231 (6)	−0.0594 (2)	0.036 (1)
O3	0.2302 (1)	0.5929 (6)	−0.0847 (2)	0.062 (1)
C1″	0.3612 (1)	0.6836 (5)	0.0460 (2)	0.030 (1)
O4	0.4053 (1)	0.8807 (5)	0.0824 (2)	0.035 (1)
C3″	0.4679 (1)	0.8398 (6)	0.2068 (2)	0.032 (1)
O5	0.5147 (1)	0.9730 (5)	0.2651 (2)	0.047 (1)
C4″	0.4634 (1)	0.6097 (6)	0.2440 (2)	0.032 (1)
C4″*a*	0.5154 (2)	0.5471 (7)	0.3962 (3)	0.048 (1)
C5″	0.4783 (1)	0.4897 (6)	0.1211 (3)	0.042 (1)
C6″	0.4058 (1)	0.5361 (6)	−0.0155 (3)	0.043 (1)
C7″	0.3744 (1)	0.5907 (6)	0.1969 (2)	0.030 (1)
C7″*a*	0.3409 (2)	0.7326 (7)	0.2833 (3)	0.046 (1)
C7″*b*	0.3462 (2)	0.3642 (6)	0.1976 (3)	0.045 (1)
Diastereoisomer (2)				
C1	0.9186 (3)	0.360	0.2211 (8)	0.047 (4)
C2	0.8704 (3)	0.538 (2)	0.1612 (7)	0.039 (4)
C3	0.8489 (5)	0.649 (2)	0.2953 (9)	0.053 (5)
C4	0.8745 (3)	0.514 (2)	0.4173 (7)	0.043 (4)
C5	0.8650 (4)	0.529 (2)	0.5587 (9)	0.059 (5)
C6	0.8953 (5)	0.387 (2)	0.6555 (9)	0.067 (6)
C7	0.9355 (5)	0.223 (2)	0.6103 (9)	0.065 (6)
C8	0.9462 (4)	0.203 (2)	0.4709 (8)	0.049 (4)
C9	0.9155 (3)	0.347 (2)	0.3747 (7)	0.037 (4)
O1	0.9536 (3)	0.253 (2)	0.1487 (6)	0.064 (4)
C1′	0.8058 (3)	0.459 (2)	0.0615 (8)	0.038 (4)
C3′	0.9087 (5)	0.686 (2)	0.0601 (9)	0.056 (5)
C4′	0.8475 (4)	0.760 (2)	−0.0429 (7)	0.049 (4)
C5′	0.8436 (5)	0.930 (2)	−0.1318 (8)	0.056 (5)
C6′	0.7824 (5)	0.968 (2)	−0.2173 (9)	0.071 (6)
C7′	0.7232 (5)	0.834 (2)	−0.2231 (9)	0.071 (6)
C8′	0.7260 (4)	0.662 (2)	−0.1346 (8)	0.057 (5)
C9′	0.7883 (3)	0.631 (2)	−0.0454 (7)	0.041 (4)
O2	0.7415 (2)	0.419 (1)	0.1319 (5)	0.046 (3)
C	0.7387 (4)	0.235 (2)	0.1996 (7)	0.042 (4)
O3	0.7857 (3)	0.107 (2)	0.1992 (7)	0.064 (3)
C1″	0.6722 (4)	0.207 (2)	0.2688 (7)	0.044 (4)
O4	0.6392 (3)	0.405 (1)	0.2993 (6)	0.058 (3)
C3″	0.5690 (4)	0.364 (2)	0.3144 (9)	0.057 (5)
O5	0.5269 (4)	0.494 (2)	0.3398 (8)	0.094 (5)
C4″	0.5574 (4)	0.132 (2)	0.2973 (8)	0.048 (4)
C4″*a*	0.4796 (7)	0.070 (2)	0.274 (2)	0.078 (8)
C5″	0.6023 (7)	0.050 (2)	0.433 (1)	0.067 (6)
C6″	0.6803 (5)	0.093 (2)	0.411 (1)	0.068 (6)
C7″	0.6089 (4)	0.096 (2)	0.1827 (8)	0.043 (4)
C7″*a*	0.5855 (5)	0.193 (2)	0.0381 (9)	0.065 (6)
C7″*b*	0.6242 (6)	−0.135 (2)	0.155 (1)	0.071 (6)

Discussion. The proposed structures of the camphanic esters (1) and (2) (Dynesen, 1976) have been verified by X-ray crystallographic analysis and are shown in Figs. 1 and 2. In both diastereomers the hydroxyl group points away from the ketone group as a result of the stereoselective reduction of the diketone.

The observation that (1) is less soluble and has a higher melting point than (2) (450–456 and 438–446 K, respectively) could indicate a more ideal packing of (1). This is confirmed by the fact that the more stable (1) has a smaller cell volume [1097.1 (1) Å^3] than (2) [1131.0 (2) Å^3]. It has been difficult to establish a simple explanation for the origin of these differences. An obvious explanation would be a different network of hydrogen bonds in the two stereoisomers. We only noted a minor difference in the O3···C3 intermolecular distance [(1): 3.178 (4) and (2): 3.29 (1) Å]. All other intermolecular C—H···O distances were of the order of 3.5 Å or greater. According to Hamilton & Ibers (1968) a methyl hydrogen to oxygen bond may be present when the C—H···O distance is approximately 3.00–3.22 Å. Thus, (1) may be stabilized more than (2) as a result of H···O interaction.

The observed differences could also be related to different van der Waals interactions. We calculated all heavy-atom interatomic distances less than 3.6 Å. A general trend of shorter intermolecular distances in (1) compared to (2) would indicate stronger van der Waals interactions. Such a trend was not found to be significant in our study. In fact, both intermolecular and intramolecular distances were strikingly similar for both compounds.

If we assume that the conformation in the solid state and in solution is the same, it is possible to

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters and bonds have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55880 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1039]

Table 2. *Bond distances* (Å) *and selected angles* (°) *with e.s.d.'s in parentheses for diastereoisomers* (1) *and* (2)

	(1)	(2)
C1—O1	1.211 (3)	1.22 (1)
C1—C2	1.533 (4)	1.53 (1)
C1—C9	1.483 (3)	1.46 (1)
C2—C3	1.544 (4)	1.55 (1)
C2—C1′	1.556 (3)	1.53 (1)
C2—C3′	1.554 (4)	1.58 (1)
C3—C4	1.509 (4)	1.48 (1)
C4—C5	1.387 (4)	1.37 (1)
C4—C9	1.390 (4)	1.41 (1)
C5—C6	1.384 (5)	1.38 (1)
C6—C7	1.382 (6)	1.39 (1)
C7—C8	1.380 (4)	1.36 (1)
C8—C9	1.388 (4)	1.38 (1)
C1′—O2	1.450 (3)	1.46 (1)
C1′—C9′	1.502 (3)	1.52 (1)
C3′—C4′	1.509 (4)	1.49 (1)
C4′—C5′	1.379 (4)	1.39 (1)
C4′—C9′	1.385 (3)	1.38 (1)
C5′—C6′	1.387 (4)	1.34 (1)
C6′—C7′	1.380 (5)	1.40 (1)
C7′—C8′	1.396 (5)	1.39 (1)
C8′—C9′	1.386 (3)	1.37 (1)
O2—C	1.330 (3)	1.36 (1)
C—O3	1.196 (3)	1.20 (1)
C—C1″	1.512 (3)	1.47 (1)
C1″—O4	1.463 (3)	1.47 (1)
C1″—C6″	1.530 (3)	1.53 (1)
C1″—C7″	1.547 (3)	1.53 (1)
O4—C3″	1.371 (3)	1.35 (1)
C3″—O5	1.197 (3)	1.19 (1)
C3″—C4″	1.511 (3)	1.53 (1)
C4″—C4″*a*	1.513 (3)	1.49 (2)
C4″—C5″	1.556 (4)	1.54 (2)
C4″—C7″	1.562 (3)	1.54 (1)
C5″—C6″	1.545 (4)	1.51 (2)
C7″—C7″*a*	1.536 (4)	1.52 (1)
C7″—C7″*b*	1.527 (4)	1.55 (1)
C2—C1—C9	107.7 (2)	108.9 (7)
C2—C1—O1	125.2 (2)	123.6 (7)
O1—C1—C9	127.1 (2)	127.5 (8)
C1—C2—C3	105.4 (2)	104.1 (6)
C1—C2—C1′	111.0 (2)	111.1 (6)
C1—C2—C3′	113.3 (2)	113.5 (6)
C1′—C2—C3	112.9 (2)	113.9 (6)
C1′—C2—C3′	101.8 (2)	101.9 (6)
C3—C2—C3′	112.6 (2)	112.7 (7)
C2—C3—C4	104.8 (2)	105.6 (7)
C3—C4—C5	128.6 (3)	130.6 (8)
C3—C4—C9	111.9 (2)	111.7 (7)
C5—C4—C9	119.4 (3)	117.7 (8)
C4—C5—C6	118.7 (3)	120.9 (8)
C5—C6—C7	121.3 (3)	120.1 (8)
C6—C7—C8	120.8 (3)	120.7 (9)
C7—C8—C9	117.8 (3)	118.6 (8)
C1—C9—C4	109.5 (2)	108.8 (7)
C1—C9—C8	128.6 (2)	129.2 (7)
C4—C9—C8	122.0 (2)	122.0 (7)
O2—C1′—C2	116.0 (2)	113.9 (6)
O2—C1′—C9′	107.8 (2)	108.2 (6)
C2—C1′—C9′	103.0 (2)	104.9 (6)
C2—C3′—C4′	103.0 (2)	103.2 (7)
C3′—C4′—C5′	130.3 (2)	129.5 (8)
C3′—C4′—C9′	109.7 (2)	111.3 (8)
C5′—C4′—C9′	120.0 (2)	119.2 (8)
C4′—C5′—C6′	119.2 (3)	119.7 (9)
C5′—C6′—C7′	120.9 (3)	121 (1)
C6′—C7′—C8′	120.3 (3)	120.1 (9)
C7′—C8′—C9′	118.2 (3)	117.4 (9)
C1′—C9′—C4′	110.2 (2)	109.0 (7)
C1′—C9′—C8′	128.3 (2)	128.6 (7)
C4′—C9′—C8′	121.4 (2)	122.4 (8)
C1′—O2—C	116.4 (2)	116.7 (6)
O2—C—O3	124.5 (2)	122.5 (7)
O2—C—C1″	113.1 (2)	113.3 (7)
O3—C—C1″	122.3 (2)	124.1 (8)
C—C1″—O4	111.1 (2)	111.6 (6)
C—C1″—C6″	112.7 (2)	116.5 (7)
C—C1″—C7″	118.9 (2)	116.9 (6)
O4—C1″—C6″	105.8 (2)	104.6 (7)
O4—C1″—C7″	102.5 (2)	101.9 (6)
C6″—C1″—C7″	104.6 (2)	103.6 (7)
C1″—O4—C3″	105.9 (2)	106.1 (6)
O4—C3″—C4″	107.2 (2)	107.9 (7)
O4—C3″—O5	121.8 (2)	123.0 (8)
C4″—C3″—O5	131.0 (2)	129.1 (8)
C3″—C4″—C4″*a*	115.0 (2)	113.8 (8)
C3″—C4″—C5″	103.3 (2)	101.2 (8)
C3″—C4″—C7″	98.8 (2)	97.6 (7)
C4″*a*—C4″—C5″	115.0 (2)	117 (1)
C4″*a*—C4″—C7″	119.4 (2)	121.5 (9)
C5″—C4″—C7″	102.7 (2)	102.2 (8)
C4″—C5″—C6″	103.9 (2)	105.2 (9)
C1″—C6″—C5″	101.2 (2)	101.9 (8)
C1″—C7″—C4″	91.1 (2)	93.1 (6)
C1″—C7″—C7″*a*	112.3 (2)	113.9 (7)
C1″—C7″—C7″*b*	115.7 (2)	113.7 (7)
C4″—C7″—C7″*a*	114.0 (2)	115.5 (7)
C4″—C7″—C7″*b*	113.9 (2)	113.9 (7)
C7″*a*—C7″—C7″*b*	109.1 (2)	106.6 (6)
Dihedral angle	91.0 (1)	91.4 (4)

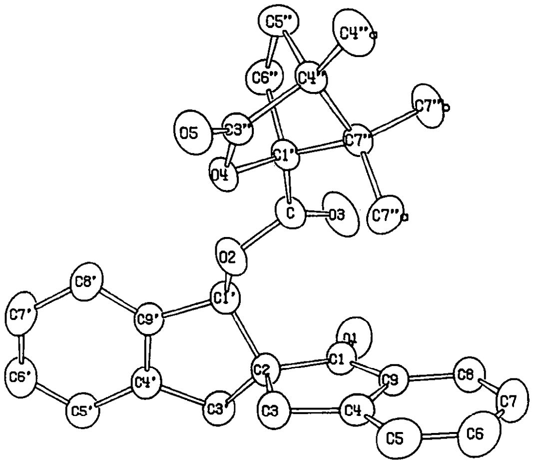


Fig. 1. *ORTEP* drawing of (1) showing the atom-numbering scheme and 50% ellipsoids.

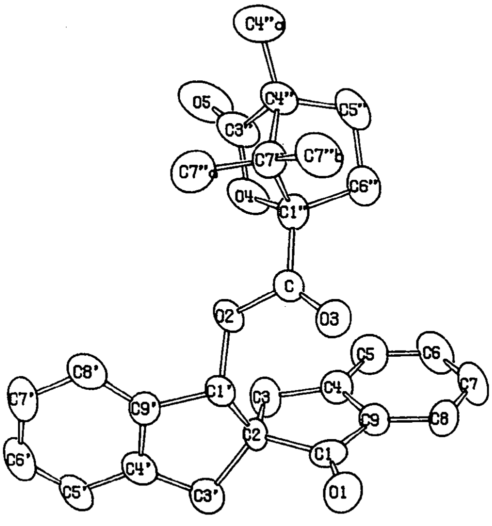


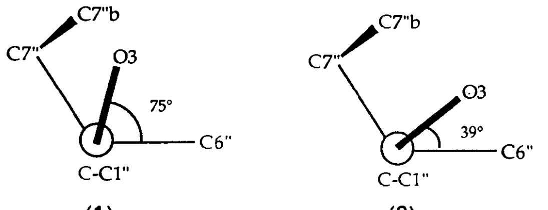
Fig. 2. *ORTEP* drawing of (2) showing the atom-numbering scheme and 50% ellipsoids.

Table 3. *Pertinent observed distances* (Å), *angles* (°) *and chemical shifts* (*p.p.m.*) *in the two diastereoisomers* (1) *and* (2) *with e.s.d.'s in parentheses*

	O3—C6″	O3—C7″*b*	O3—C—C1″—C6″	δC6″	δC7″*b*	δH(C7″*b*)
(1)	3.178 (4)	3.125 (3)	74.5 (3)	30.471	15.763	0.53
(2)	2.96 (1)	3.37 (1)	39 (1)	30.137	16.091	0.81

explain some typical differences in chemical shifts in the observed ^1H and ^{13}C NMR spectra by comparing the intramolecular distances of the stereoisomers as found in the crystallographic study. Pertinent observed geometry and chemical shifts are given in Table 3. The C7″*b*—O3 and C6″—O3 interactions are the only conformational differences in the two stereoisomers which may conceivably influence the chemical shifts δC6″, δC7″*b* and δH(C7″*b*). In (1), the carbonyl O atom of the camphanic ester moiety points in the direction of C7″*b*, whereas in (2) it is oriented more towards C6″, as can be seen from Fig. 3.

A carbonyl group affects the chemical shifts of neighbouring nuclei (Williams & Fleming, 1987). We observe a correlation between the chemical shift values of a given nucleus and the distance from the nucleus to the carbonyl O atom. The structure with a C or an H atom closer to the carbonyl O atom gives a chemical shift at higher field. In (1), both H(C7″*b*) and C7″*b* are shifted to higher field, whereas in (2) C6″ is shifted upfield. Thus, it seems likely that the conformations of the two esters observed in the solid state are preserved in solution. The different intramolecular interactions also result in another difference in the ^1H spectra. The C5″ and C6″ methylene protons in (1) show a characteristic *ABCD* pattern, whereas in (2) the *ABCD* coupling system is influenced by the carbonyl group, resulting in a complicated coupling system. The trend is that this new coupling system is found at higher field. The reason for the different twisting of the camphanic esters around the C—C1″ bond is as yet unknown, but the crystallographic study corroborates the conclusions drawn from the NMR studies.



(1) (2)

Fig. 3. Newman projection along the C—C1″ bond. Only the relevant atoms are shown.

Thus, the X-ray crystallographic study verifies the configurations of the two camphanic esters. NMR studies support the results found in the crystallographic study. In spite of considerable differences in physical properties, such as solubility and melting point, relatively small differences in interatomic distances are observed.

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Structure of 4-Iodo-*trans*-cinnamic Acid and a Study of Carboxyl Group Disorder

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Abstract. (*E*)-3-(4-Iodophenyl)propenoic acid, $C_9H_7IO_2$, $M_r = 274.05$, monoclinic, $P2_1/n$, $a = 4.118$ (2), $b = 6.274$ (4), $c = 34.672$ (2) Å, $\beta = 90.32°$, $V = 895.7$ (9) Å^3, $Z = 4$, $D_x = 2.03$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 34.90$ cm^{-1}, $F(000) = 520$, $T = 153$ K, $R = 0.037$, $wR = 0.043$ for 1098

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