Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. de York, Angleterre, et Louvain-la-Neuve, Belgique.

Motherwell, S. & Clegg, B. (1978). *PLUTO.* Programme pour le dessin des structures moléculaires et cristallines.

Sheldrick, G. M. (1976). *SHELX76.* Programme pour la détermination des structures cristallines. Univ. de Cambridge, Angleterre.

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2-{8-Hydroxybicyclo[5.1.0]oct-1(7)-en-8-yl}-1-cycloheptene-1-carboxylic Acid γ-Lactone, $C_{16}H_{20}O_2$ (I), and 2-{(1α,7α,8α)-8-Hydroxybicyclo[5.1.0]oct-8-yl}-1-cycloheptene-1-carboxylic Acid γ-Lactone, $C_{16}H_{22}O_2$ (II)

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Abstract. (I) $M_r = 244{\cdot}3$, monoclinic, $P2_1/c$, $a = 8{\cdot}994\ (1)$, $b = 14{\cdot}955\ (2)$, $c = 9{\cdot}863\ (2)$ Å, $\beta = 93{\cdot}76\ (2)°$, $V = 1324$ Å^3, $Z = 4$, $F(000) = 528$, $D_m = 1{\cdot}22$, $D_x = 1{\cdot}23$ g cm^{-3}, $\mu = 5{\cdot}8$ cm^{-1}, $R = 8{\cdot}3\%$ for 1080 reflections having $F > 2\sigma_F$. (II) $M_r = 246{\cdot}3$, tetragonal, $P4_12_12$, $a = 7{\cdot}961\ (2)$, $c = 42{\cdot}919\ (1)$ Å, $V = 2720$ Å^3, $Z = 8$, $F(000) = 1072$, $D_m = 1{\cdot}23$, $D_x = 1{\cdot}20$ g cm^{-3}, $\mu = 5{\cdot}73$ cm^{-1}, $R = 5{\cdot}2\%$ for 1607 reflections having $F > 2\sigma_F$. All data were collected at 298 K using Cu $K\alpha$ radiation ($\bar{\lambda} = 1{\cdot}5418$ Å). Compound (II) is the hydrogenation product of compound (I) in which the added H atoms are shown to be on the same side of the cyclopropyl ring as the lactone group. The connectivity of the compounds agrees with prediction based upon chemical and spectroscopic evidence.

Introduction. In the course of the synthesis of functionalized macrocycles, compound (I) and its hydrogenation product (II) were prepared (Finnegan & Delecki, 1969). X-ray analysis was undertaken in order to determine the connectivity of both molecules and the stereochemistry of the hydrogenation product.

Experimental. (I) Crystallized from ethanol. Crystal $0{\cdot}08 \times 0{\cdot}08 \times 0{\cdot}40$ mm. Lattice parameters determined using 27 reflections, $40° < 2\theta < 58°$. Systematic absences $h0l$ $l = 2n + 1$, $0k0$ $k = 2n + 1$. Crystal mounted with **a** parallel to φ axis. Data with $\theta < 53°$ measured. Max. variation in intensity standards 3%.

Table 1. *Atomic coordinates* [$\times 10^5$ *for z in* (II), $\times 10^4$ *for all others*] *and isotropic thermal parameters* ($\times 10$) *for nonhydrogen atoms, with e.s.d.'s in parentheses*

The temperature factor is of the form $\exp[-B(\sin\theta/\lambda)^2]$.

	x	y	z	B(Å^2)
Compound (I)				
C(1)	2880 (6)	−502 (4)	2015 (5)	39 (2)
C(2)	2564 (7)	−1477 (4)	2003 (6)	43 (2)
C(3)	1676 (7)	−1697 (4)	672 (7)	48 (2)
C(4)	2278 (7)	1294 (4)	623 (6)	47 (2)
C(5)	1805 (7)	−355 (4)	1005 (6)	40 (2)
C(6)	2687 (7)	385 (4)	−271 (5)	40 (2)
C(7)	2909 (6)	162 (4)	1185 (5)	37 (2)
C(8)	3555 (6)	334 (3)	2602 (5)	33 (2)
C(9)	3008 (5)	945 (3)	3635 (5)	30 (1)
C(10)	1383 (6)	1090 (5)	3784 (6)	39 (2)
C(11)	921 (7)	1081 (4)	5231 (6)	42 (2)
C(12)	1498 (7)	1846 (5)	6139 (7)	48 (2)
C(13)	3155 (7)	1822 (5)	6558 (6)	43 (2)
C(14)	4178 (6)	2015 (4)	5431 (6)	39 (2)
C(15)	4162 (5)	1321 (3)	4339 (5)	28 (1)
C(16)	5537 (7)	958 (4)	3839 (5)	40 (2)
O(1)	5172 (4)	366 (3)	2805 (3)	41 (1)
O(2)	6820 (4)	1101 (3)	4210 (4)	53 (1)
Compound (II)				
C(1)	339 (3)	1323 (3)	4613 (4)	32 (1)
C(2)	−1392 (3)	561 (3)	5036 (5)	41 (1)
C(3)	−2497 (3)	873 (4)	2165 (5)	54 (1)
C(4)	3254 (3)	2655 (4)	1996 (5)	56 (1)
C(5)	2049 (3)	4107 (4)	1543 (5)	49 (1)
C(6)	−852 (3)	4410 (3)	4301 (4)	41 (1)
C(7)	588 (3)	3189 (3)	4250 (4)	33 (1)
C(8)	1321 (2)	2282 (2)	7068 (4)	28 (1)
C(9)	976 (2)	2554 (2)	10422 (4)	27 (1)
C(10)	703 (2)	2923 (3)	11864 (4)	32 (1)
C(11)	1099 (3)	1879 (3)	14753 (5)	42 (1)
C(12)	−79 (3)	2325 (4)	17663 (5)	49 (1)
C(13)	1742 (3)	1746 (3)	17587 (5)	43 (1)
C(14)	2855 (3)	2689 (3)	15324 (5)	39 (1)
C(15)	2446 (2)	2471 (2)	11949 (4)	30 (1)
C(16)	3790 (2)	2162 (2)	9696 (4)	32 (1)
O(1)	3091 (2)	2034 (2)	6801 (3)	34 (1)
O(2)	5284 (2)	2022 (2)	10070 (4)	46 (1)

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1565 unique reflections measured. Final $R = 0{\cdot}083$ for 1080 reflections with $F > 2\sigma_F$, $R_w = 0{\cdot}069$, $S = 1{\cdot}28$, $(\Delta/\sigma)_{\max} = 0{\cdot}30$ and $(\Delta/\sigma)_{\rm ave} = 0{\cdot}05$. Final difference map had no peaks $>0{\cdot}4$ e Å^{-3}. (II) Crystallized from absolute ethanol. Crystal $0{\cdot}4 \times 0{\cdot}6 \times 0{\cdot}6$ mm mounted with **a** parallel to φ axis. Lattice parameters determined using 33 reflections, $80° < 2\theta < 90°$. Conditions for reflections ($00l$ $l = 4n$ present, $h00$ $h = 2n$ present) indicated space group $P4_12_12$ or $P4_32_12$; $P4_12_12$ was chosen arbitrarily in the absence of atoms with sufficient anomalous-scattering power to permit distinction. Max. variation in intensity standards 3%. 1747 unique reflections with $\theta < 75°$ measured. Final $R = 0{\cdot}052$ for 1607 reflections with $F > 2\sigma_F$, $R_w = 0{\cdot}064$, $S = 1{\cdot}78$, $(\Delta/\sigma)_{\max} = 0{\cdot}45$ and $(\Delta/\sigma)_{\rm ave} = 0{\cdot}05$. Final difference map had no peaks $>0{\cdot}25$ e Å^{-3}.

For each structure: Density measured by flotation in KI solution. Lattice parameters determined by least-squares procedure using centered 2θ values. Intensity data collected on a CAD-4 diffractometer, θ–2θ scan technique, Ni-filtered Cu $K\alpha$ radiation. Two standard reflections measured after each 100 intensity measurements; there was intensity fluctuation but no evidence of crystal decay. No absorption correction. Phase determination by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971). Least-squares refinement on $|F|$. All H atoms in both structures located in difference maps. Positional parameters for all atoms and anisotropic thermal parameters for nonhydrogen atoms were refined. The H atoms in (II) were refined isotropically and those in (I) were assigned isotropic thermal parameters based upon the motion of the C atom to which they are bonded. $w = 1/\sigma_F^2$ where σ_F was defined by Stout & Jensen (1968, equation H.14) with instability correction 0·06. No secondary-extinction correction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Table 2.2B). Computer programs used included the Enraf–Nonius *Structure Determination Package*, *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and *ORTEP* (Johnson, 1965).

Discussion. The atomic positional and thermal parameters are given in Table 1. The atomic numbering, bond distances, bond angles and torsion angles involving nonhydrogen atoms are presented in Fig. 1. The

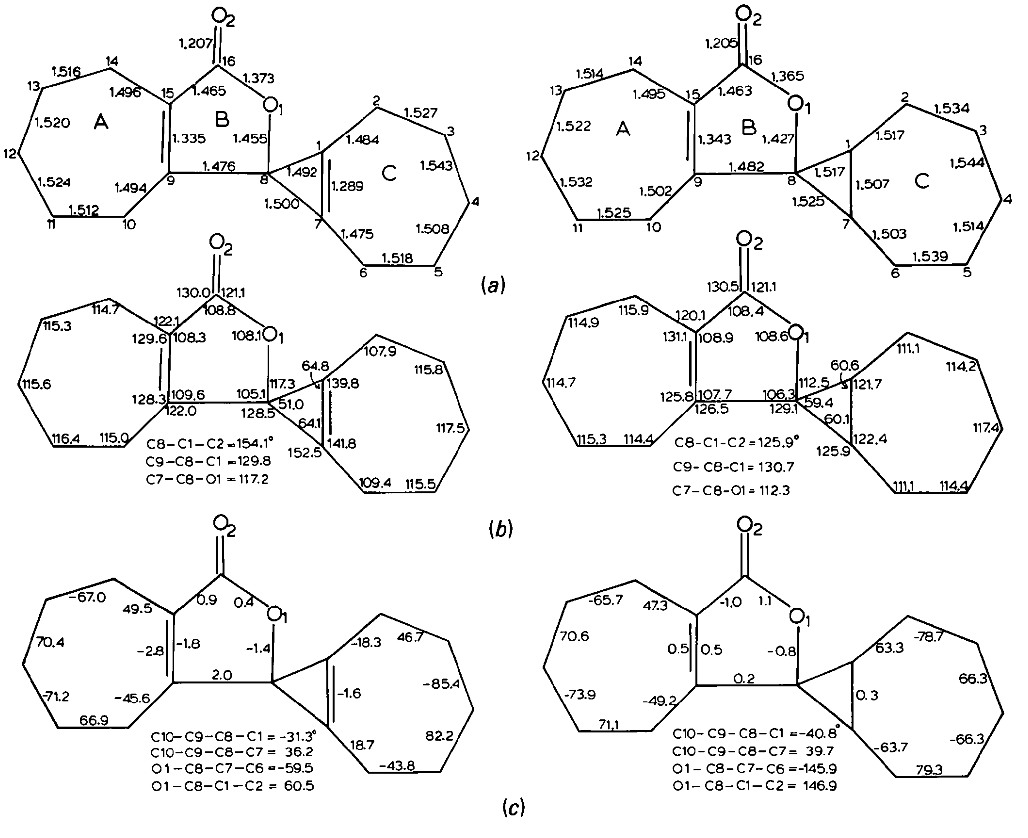

Fig. 1. (*a*) Bond lengths (Å), (*b*) selected bond angles (°) and (*c*) torsion angles (°) for (I) and (II). The σ ranges for bond lengths, bond angles and torsion angles are 0·006–0·009 Å, 0·3–0·5°, and 0·5–1·0° for compound (I) (left) and 0·002–0·004 Å, 0·1–0·2° and 0·2–0·3° for compound (II) (right).

observed C—H distances are in the ranges (I) 0·95–1·10 Å and (II) 0·87–1·12 Å.*

The connectivity of the compounds revealed by crystallographic analysis agrees with the predictions based upon chemical and spectroscopic evidence (Delecki, 1970). In the hydrogenation product (II), the H atoms were added to C(1) and C(7) on the same side of the cyclopropyl ring as the O(1) substituent (Fig. 2). The bond lengths and angles in the *A* rings of the two structures are in excellent agreement, and the overall conformations of the rings are nearly identical despite differences in crystal packing. A least-squares fit of atoms C(8) to C(16), O(1) and O(2) in the two structures yields a mean-square difference in the relative position of these atoms of 0·03 Å. The *A* rings have a chair conformation in which atoms C(9) and C(15) are 'above' and C(12) is 'below' the plane of atoms C(10), C(11), C(13) and C(14). The ring has mirror symmetry across a plane bisecting the C(9)—C(15) bond and intersecting C(12). The *C* ring of (II) has a similar chair conformation with atoms C(1) and C(7) 'above' and atom C(4) 'below' the plane of atoms C(2), C(3), C(5) and C(6). This ring is more symmetric and more puckered than the *A* rings as indicated by the magnitudes of its torsion angles. The *C* ring of (I) also has a chair conformation with a mirror plane intersecting C(4) and bisecting the C(1)—C(7) bond. This ring however is much flatter due to the double bond in the cyclopropyl ring. The positions of C(1), C(7) and C(4) relative to the other four atoms of the *C* ring in (I) are reversed as indicated by the change of signs of the torsion angles from those of the *C* ring of (II). There are no intermolecular contacts less than 3·35 Å between nonhydrogen atoms.

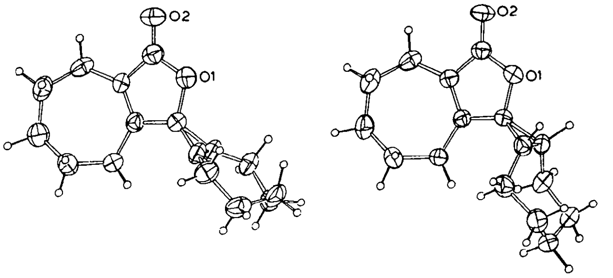

Fig. 2. *ORTEP* drawings of (I) (left) and (II) (right) with thermal ellipsoids for nonhydrogen atoms scaled to 50% probability.

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References

Delecki, D. J. (1970). MS Thesis. Department of Medicinal Chemistry, State Univ. of New York at Buffalo.

Finnegan, R. A. & Delecki, D. J. (1969). *Abstracts of Papers.* 158th Natl Meet. Am. Chem. Soc., New York. ORGN79.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Johnson, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Stout, G. H. & Jensen, L. H. (1968). *X-ray Structure Determination.* New York: Macmillan.

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* Tables of structure factors, positional and thermal parameters for H, anisotropic thermal parameters for the non-H atoms and packing diagrams have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39379 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Low-Temperature X-ray Study of Thianthrene 5,5,10,10-Tetraoxide, $C_{12}H_8O_4S_2$

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Abstract. $M_r = 280·33$, orthorhombic, $Pbcn$, $a = 19·613\,(9)$, $b = 12·161\,(2)$, $c = 14·433\,(8)$ Å, $V = 3442\,(3)$ Å^3, $Z = 12$, $D_m = 1·59\,(2)$ (294 K), $D_x = 1·623$ g cm^{-3} (163 K), $\lambda(\text{Mo}\,K\alpha_{1,2}) = 0·71069$ Å, $\mu = 4·57$ cm^{-1}, $F(000) = 1728$. Final $R = 0·050$ for 3077 observed reflections. There are 1·5 molecules in the asymmetric unit. In one unit cell, there are eight molecules located on general position *d*, while four molecules lie around twofold axes in such a way that the S···S intramolecular axes are perpendicular to the

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