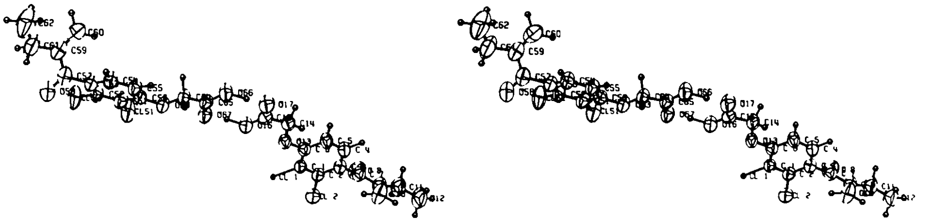

Fig. 2. Vue stéréoscopique de la conformation.

l'hypothèse d'une même conformation, représentée dans la Fig. 2.

Le cycle benzène de la molécule *A* forme un plan contenant O(13) et Cl(1) dans la limite des déviations standard, et Cl(2) et C(7) à 0,1 Å près. Les atomes C(7), O(13), Cl(1) et Cl(2) de la molécule *B* sont distants de moins de 0,1 Å du plan du cycle benzène de cette molécule.

Le Tableau 4 rassemble les distances intermoléculaires inférieures à 3,25 Å. On remarquera le contact Cl(1)–Cl(1) de 3,217 Å et les liaisons hydrogène courtes entre les deux molécules de l'unité asymétrique, formant des angles O(16)–H–O(17) de 173°. La Fig. 3* montre une vue stéréoscopique de l'empilement des molécules dans la maille. L'angle entre les plans C(15)–O(16)–O(17) des molécules *A* et *B* est de 6°.

Les auteurs remercient MM les Professeurs J. Toussaint et C. L. Lapière pour l'intérêt qu'ils ont porté à ce travail.

Références

Ahmed, F. R., Hall, S. R., Pippy, M. E. & Saunderson, C. P. (1967). NRC Crystallographic Programs for the IBM 360 System. National Research Council, Ottawa, Canada.

Declercq, J. P., Germain, G., Main, P. & Woolfson, M. M. (1973). *Acta Cryst.* A29, 231–234.

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* La Fig. 3 a été déposée. Voir la note précédente.

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Dimethyl 6′,7′-Dimethyl-3′-oxospiro[oxirane-2,4′-tricyclo[3.3.0.0^{2,8}]oct[6]ene]-1′,8′-dicarboxylate

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Abstract. $C_{15}H_{16}O_6$, $M_r = 292{\cdot}3$, monoclinic $P2_1/a$, $Z = 4$, $a = 14{\cdot}714$ (6), $b = 6{\cdot}133$ (2), $c = 17{\cdot}462$ (7) Å, $\beta = 112{\cdot}5$ (1)°. The structure was refined to an R of 0·036 for 1307 observed reflexions. The structure proposed from other evidence is confirmed. There are no unusually short intermolecular distances.

Introduction. The colourless, compact prismatic crystals were supplied by Dr H.-D. Becker (Department of Organic Chemistry, University of Gothenburg). A specimen 0·4 × 0·6 × 0·4 mm was mounted on a Syntex $P2_1$ diffractometer. A θ–2θ time-variable scan technique was used, $2\theta_{\max}$ being 100°, with a scan speed varying between 3·0 and 30·0° min^{-1}. Graphite-monochromatized Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å) was used to measure 1476 reflexions, 1307 of which were retained when an observed–unobserved cut-off at $4{\cdot}0\sigma(I)$ [$\sigma(I)$ from counting statistics] was employed. The systematic absences, $0k0$, $k = 2n + 1$ and $h0l$, $h = 2n + 1$, uniquely determine the space group as $P2_1/a$. No absorption correction was made.

The structure was solved with *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1974) from 200 E values, 1600 $\sum_2$ relations and 16 sets of phases, and refined with anisotropic temperature factors for the non-hydrogen atoms. The final R value was 0·036 for

Table 1. *Positional* ($\times 10^4$, *except* $\times 10^3$ *for* H) *and, for* H *atoms, isotropic thermal parameters*

	x	*y*	*z*	*U* (Å^2)
O(1)	8465 (1)	7770 (4)	8438 (1)	
O(2)	8588 (2)	4403 (4)	8963 (1)	
O(3)	6641 (2)	738 (4)	8464 (1)	
O(4)	6305 (2)	3925 (4)	8917 (1)	
O(5)	6981 (1)	1549 (3)	5918 (1)	
O(6)	7918 (1)	5955 (4)	5985 (1)	
C(1)	7377 (2)	5158 (5)	7635 (2)	
C(2)	7229 (2)	2856 (5)	7288 (2)	
C(3)	7053 (2)	3035 (5)	6389 (2)	
C(4)	7085 (2)	5430 (5)	6196 (2)	
C(5)	6934 (2)	6691 (5)	6882 (2)	
C(6)	5836 (2)	6657 (5)	6691 (2)	
C(7)	5604 (2)	5000 (5)	7077 (2)	
C(8)	6513 (2)	3823 (5)	7634 (1)	
C(9)	8209 (2)	5693 (5)	8415 (2)	
C(10)	9229 (3)	8563 (7)	9201 (2)	
C(11)	6490 (2)	2860 (5)	8418 (2)	
C(12)	6607 (3)	−338 (6)	9199 (2)	
C(13)	6941 (2)	6184 (6)	5365 (2)	
C(14)	5170 (2)	8196 (6)	6061 (2)	
C(15)	4603 (2)	4254 (6)	7004 (2)	
H(21)	762 (1)	177 (4)	757 (1)	0·029 (6)
H(51)	725 (2)	809 (4)	699 (1)	0·038 (7)
H(101)	976 (3)	766 (7)	932 (2)	0·142 (15)
H(102)	902 (3)	835 (6)	964 (2)	0·121 (14)
H(103)	946 (4)	990 (9)	906 (3)	0·181 (21)
H(121)	716 (3)	031 (8)	967 (3)	0·157 (19)
H(122)	596 (3)	1012 (7)	923 (2)	0·126 (14)
H(123)	654 (4)	813 (9)	906 (3)	0·171 (21)
H(131)	676 (2)	504 (5)	490 (2)	0·057 (8)
H(132)	673 (2)	760 (4)	522 (1)	0·042 (7)
H(141)	456 (2)	806 (6)	601 (2)	0·090 (11)
H(142)	514 (3)	769 (7)	549 (2)	0·136 (15)
H(143)	537 (3)	975 (8)	615 (2)	0·148 (17)
H(151)	448 (3)	268 (8)	682 (2)	0·142 (17)
H(152)	457 (2)	401 (5)	754 (2)	0·091 (11)
H(153)	408 (2)	524 (5)	666 (2)	0·075 (10)

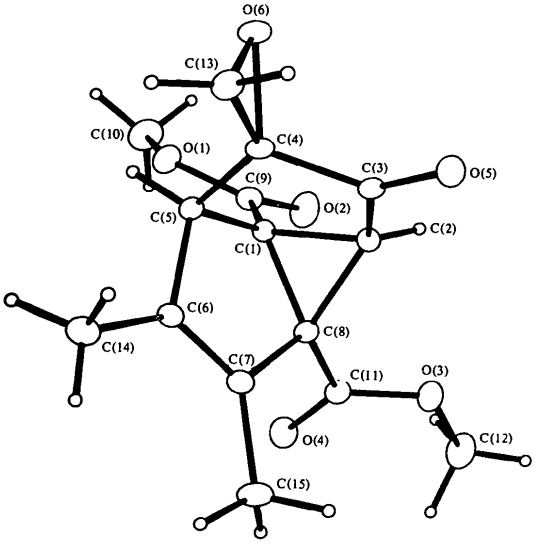

Fig. 1. A molecule of $C_{15}H_{16}O_6$ showing the atom-numbering scheme.

Table 2. *Bond lengths* (Å)

C(1)–C(2)	1·518 (4)	C(11)–O(3)	1·318 (4)
C(1)–C(5)	1·543 (4)	C(11)–O(4)	1·201 (4)
C(1)–C(8)	1·512 (4)	C(12)–O(3)	1·461 (4)
C(1)–C(9)	1·478 (4)	C(13)–O(6)	1·437 (4)
C(2)–C(3)	1·494 (4)	C(2)–H(21)	0·89 (2)
C(2)–C(8)	1·521 (4)	C(5)–H(51)	0·96 (2)
C(3)–C(4)	1·511 (4)	C(10)–H(101)	0·92 (4)
C(3)–O(5)	1·206 (3)	C(10)–H(102)	0·95 (4)
C(4)–C(5)	1·512 (4)	C(10)–H(103)	0·95 (5)
C(4)–O(6)	1·445 (3)	C(12)–H(121)	0·99 (4)
C(4)–C(13)	1·459 (4)	C(12)–H(122)	1·01 (4)
C(5)–C(6)	1·529 (4)	C(12)–H(123)	0·97 (5)
C(6)–C(7)	1·333 (4)	C(13)–H(131)	1·03 (3)
C(6)–C(14)	1·496 (5)	C(13)–H(132)	0·92 (3)
C(7)–C(8)	1·503 (4)	C(14)–H(141)	0·87 (3)
C(7)–C(15)	1·501 (4)	C(14)–H(142)	1·04 (4)
C(8)–C(11)	1·503 (4)	C(14)–H(143)	0·99 (5)
C(9)–O(1)	1·324 (4)	C(15)–H(151)	1·01 (5)
C(9)–O(2)	1·202 (4)	C(15)–H(152)	0·97 (3)
C(10)–O(1)	1·459 (4)	C(15)–H(153)	0·98 (3)

Table 3. *Bond angles* (°)

C(2)–C(1)–C(5)	106·1 (2)	C(6)–C(7)–C(8)	110·9 (2)
C(2)–C(1)–C(8)	60·3 (2)	C(6)–C(7)–C(15)	128·5 (3)
C(2)–C(1)–C(9)	121·1 (2)	C(8)–C(7)–C(15)	120·6 (3)
C(5)–C(1)–C(8)	104·3 (2)	C(1)–C(8)–C(2)	60·1 (2)
C(5)–C(1)–C(9)	125·5 (2)	C(1)–C(8)–C(7)	106·3 (2)
C(8)–C(1)–C(9)	121·3 (2)	C(1)–C(8)–C(11)	121·6 (2)
C(1)–C(2)–C(3)	106·9 (2)	C(2)–C(8)–C(7)	120·6 (2)
C(1)–C(2)–C(8)	59·7 (2)	C(2)–C(8)–C(11)	117·9 (2)
C(3)–C(2)–C(8)	121·4 (2)	C(7)–C(8)–C(11)	117·0 (2)
C(2)–C(3)–C(4)	125·7 (2)	C(1)–C(9)–O(1)	111·5 (2)
C(2)–C(3)–O(5)	126·6 (3)	C(1)–C(9)–O(2)	123·7 (3)
C(4)–C(3)–O(5)	107·4 (2)	C(1)–C(9)–O(2)	124·7 (3)
C(3)–C(4)–C(5)	107·1 (2)	C(9)–O(1)–O(2)	117·3 (2)
C(13)–C(4)–O(6)	59·3 (2)	C(8)–C(11)–O(3)	112·5 (2)
C(1)–C(5)–C(4)	102·8 (2)	C(8)–C(11)–O(4)	122·5 (3)
C(1)–C(5)–C(6)	104·2 (2)	O(3)–C(11)–O(4)	124·9 (3)
C(4)–C(5)–C(6)	106·6 (2)	C(11)–O(3)–C(12)	116·0 (2)
C(5)–C(6)–C(7)	110·9 (2)	C(4)–C(13)–O(6)	59·9 (2)
C(5)–C(6)–C(14)	119·7 (2)	C(4)–O(6)–C(13)	60·8 (2)
C(7)–C(6)–C(14)	129·0 (2)		

1307 reflexions (0·042 with 169 unobserved reflexions included) when 255 parameters were refined. The scattering factors were those of Doyle & Turner (1968) for C and O and of Stewart, Davidson & Simpson (1965) for H. The final atomic positions and thermal parameters are given in Table 1.*

Discussion. Several products have been obtained by the photochemical rearrangement of bicyclo[2.2.2]octa-

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* Lists of structure factors and anisotropic thermal parameters and Fig. 3 have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33496 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

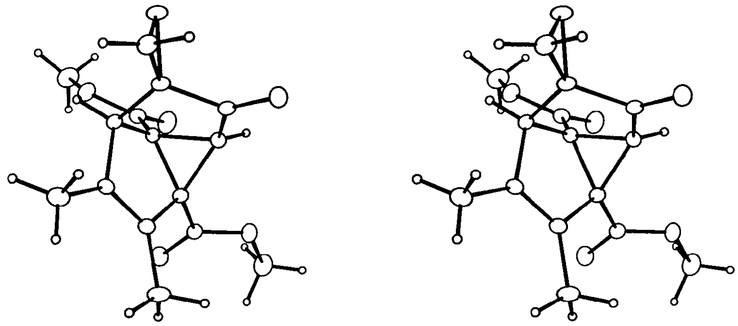

Fig. 2. Stereoview of the molecule.

Table 4. *Torsion angles in the rings* (°)

C(1)–C(2)–C(3)–C(4)	0·6	C(1)–C(5)–C(6)–C(7)	14·9
C(2)–C(3)–C(4)–C(5)	18·8	C(5)–C(6)–C(7)–C(8)	−5·1
C(3)–C(4)–C(5)–C(1)	−29·9	C(6)–C(7)–C(8)–C(1)	−7·2
C(4)–C(5)–C(1)–C(2)	30·1	C(7)–C(8)–C(1)–C(5)	15·8
C(5)–C(1)–C(2)–C(3)	−19·3	C(8)–C(1)–C(5)–C(6)	−18·3

dienones (Becker & Ruge, 1975). These authors used chemical and NMR methods to propose the structure of the products and, as a confirmation of this, a crystal structure investigation has been undertaken. The proposed structure proved to be correct.

A drawing of the content of the asymmetric unit, giving the atomic numbering scheme, is shown in Fig. 1. Intramolecular bond distances and angles are listed in Tables 2 and 3. Stereodiagrams of the structure (Johnson, 1965) are given in Figs. 2 and 3.* Table 4 shows the torsion angles of the two five-membered rings. The three-membered ring, C(4)–C(13)–O(6), is approximately normal (88·5°) to the connecting five-membered ring, the latter being defined by C(1) to C(5).

The author thanks Dr H.-D. Becker for supplying the crystals and suggesting the problem, and Dr John Wood for revising the English text. This work has been supported by the Swedish Natural Science Research Council (Contract No. 2318).

References

Becker, H.-D. & Ruge, B. (1975). *Angew. Chem.* **82**, 782–783.

Doyle, P. A. & Turner, P. S. (1968). *Acta Cryst.* A**24**, 390–397.

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

Main, P., Lessinger, L., Woolfson, M. M., Germain, G. & Declercq, J. P. (1974). *MULTAN* 74, *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. York, England, and Louvain, Belgium.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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* Fig. 3 has been deposited. See preceding footnote.

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tert-Butyloxycarbonyl-L-prolylsarcosine

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Abstract. $C_{13}H_{22}N_2O_5$, $M_r = 286{\cdot}33$, orthorhombic, $P2_12_12_1$, $a = 13{\cdot}955$ (1), $b = 16{\cdot}767$ (1), $c = 6{\cdot}662$ (1) Å, $Z = 4$, $D_m = 1{\cdot}22$, $D_x = 1{\cdot}218$ g cm^{-3}, μ(Cu $K\alpha$) = 7·44 cm^{-1}. $R = 0{\cdot}049$ for 966 reflections. The main-chain structure is similar to that of *tert*-butyloxycarbonyl-sarcosylglycine benzyl ester. The