CAMERON, T. S., AMERO, R. B. & CORDES, R. E. (1980). *Cryst. Struct. Commun.* **9**, 533–537, 539–542.
CHADHA, R. K. & DRAKE, J. E. (1984). *J. Organomet. Chem.* **268**, 141–147.
CHADHA, R. K. & MILLER, J. M. (1982). *J. Chem. Soc. Dalton Trans.* pp. 117–120.
COMASSETO, J. V. & PETRAGNANI, N. (1983). *Synth. Commun.* **13**, 889–899.
CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.
DANISHEFSKY, S., HIRAMA, M., FRITCH, N. & CLARDY, J. (1979). *J. Am. Chem. Soc.* **101**, 7013–7018.
DAVIS, M. & HASSEL, O. (1963). *Acta Chem. Scand.* **17**, 1181.
DEWAN, J. C. & SILVER, J. (1977). *Acta Cryst.* B**33**, 2671–2674.
FLIPPEN, J. L., KARLE, J. & KARLE, I. L. (1970). *J. Am. Chem. Soc.* **92**, 3749–3755.
FOSS, O. (1962). *Acta Chem. Scand.* **16**, 779–780.
FOSS, O. (1967). In *Selected Topics in Structure Chemistry*, edited by P. ANDERSON, O. BASTIENSEN & S. FURBERG, pp. 145–173. Oslo: Universitetsforlaget.
GILLESPIE, R. J. (1970). *J. Chem. Educ.* **47**, 18–23.
HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.
HARDY, A. D. U., MCKENDRICK, J. J., MACNICOL, D. D. & WILSON, D. R. (1979). *J. Chem. Soc. Perkin Trans.* 2, pp. 729–734.
HUSEBYE, S. (1983). *Proc. 4th Int. Conf. on the Organic Chemistry of Selenium and Tellurium*, edited by F. J. BERRY & W. R. MCWHINNIE, pp. 301–378. Birmingham.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
JONES, R. H. & HAMOR, T. A. (1984). *J. Organomet. Chem.* **262**, 151–155; **269**, 11–20.
MANGION, M. M., ZINGARO, R. A. & MEYERS, E. A. (1975). *Chem. Scr.* 8A, 45–50.
MURRAY-RUST, P., MURRAY-RUST, J. & NEWTON, R. F. (1979). *Acta Cryst.* B**35**, 1918–1920.
PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd. ed. Ithaca, NY: Cornell Univ. Press.
SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
SINGH, H. B., MCWHINNIE, W. R., HAMOR, T. & JONES, R. H. (1984). *J. Chem. Soc. Dalton Trans.* pp. 23–28.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
ZIOLO, R. F. & TROUP, J. (1983). *J. Am. Chem. Soc.* **105**, 229–235.

Acta Cryst. (1987). C**43**, 1151–1153

Structure of 10-(2,6-Dichlorophenyl)-8-oxa-9-aza[5.3.3]propell-9-en-2-one* (DCPOP)

BY C. A. KAVOUNIS AND P. J. RENTZEPERIS

Applied Physics Laboratory, Department of Physics, Aristotle University of Thessaloniki, Thessaloniki, Greece

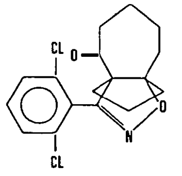
AND E. MALAMIDOU-XENIKAKI AND E. COUTOULI-ARGYROPOULOU

Laboratory of Organic Chemistry, Department of Chemistry, Aristotle University of Thessaloniki, Thessaloniki, Greece

(*Received* 12 *February* 1986; *accepted* 23 *December* 1986)

Abstract. $C_{17}H_{17}Cl_2NO_2$, $M_r = 338{\cdot}2$, monoclinic, $P2_1/c$, $a = 6{\cdot}8940\ (8)$, $b = 10{\cdot}941\ (1)$, $c = 20{\cdot}431\ (6)$ Å, $\beta = 95{\cdot}59\ (2)°$, $V = 1533{\cdot}73$ Å^3, $Z = 4$, $D_m = 1{\cdot}454$, $D_x = 1{\cdot}464$ Mg m^{-3}, m.p. = 408–410 K, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}4274$ mm^{-1}, $F(000) = 704$, $T = 298$ K, final $R = 0{\cdot}083$ for 1172 unique reflections. The mean plane of the cycloheptanone ring nearly bisects the angle formed at the common C—C bond, of length 1·55(1) Å, by the cyclopentane and dihydroisoxazole rings. The plane of the dichlorophenyl ring is almost perpendicular [84·8 (3)°] to the plane of the latter. The cycloheptanone ring shows a chair conformation.

Introduction. Propellanes are tricyclic compounds in which three rings are fused together at a common conjoining carbon–carbon bond. In these compounds it is of special importance to know the type of bonding between the two quarternary bridge-head C atoms and the resulting characteristics of the conjoining bond. In large-ring propellanes, as in our case, the hybridization of these C atoms may be sp^3 or sp^2 (Herr, 1977). The title compound was isolated during systematic preparative work on new heterocyclic propellanes using 1,3-dipolar cycloaddition of nitrile oxides to bicyclo[5.3.0]dec-1(7)-en-2-one (Malamidou-Xenikaki, Coutouli-Argyropoulou & Alexandrou, 1985). The spectroscopic and theoretical data favoured the structure (I). However, to determine the structure unambiguously an X-ray analysis was considered necessary.



(I)

---

* 10-(2,6-Dichlorophenyl)-8-oxa-9-azatricyclo[$5.3.3.0^{1,7}$]tridec-9-en-2-one.

0108-2701/87/061151-03$01.50

Experimental. Small transparent crystals, size 0·3 × 0·4 × 0·5 mm. D_m measured by flotation in KBr solution; computer-controlled Philips PW 1100 four-circle single-crystal diffractometer; graphite-monochromated Mo$K\alpha$, $\theta/2\theta$ scan mode; lattice parameters and standard deviations were calculated by least-squares analysis with *LATCON* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) of the θ angles of 31 strong reflections in the range 4–12°. Absorption correction not applied; 3923 reflections, of which 2681 independent, were measured in the range $\theta = 3$–$25°$, index range $h = -3 \rightarrow 8$, $k = -5 \rightarrow 13$, $l = -24 \rightarrow 24$; no decrease in intensity in the 3 standard reflections measured every 100 min; correction for Lorentz and polarization factors calculated with *DATRED* (Main, 1970). $R_{\text{int}} = 0·053$ from 1158 equivalent reflections, 1495 reflections were recognized as unobserved and 1172 as observed $[I > 2\sigma(I)]$. The structure was solved with *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977); refinement based on F magnitudes in full-matrix least-squares calculation with *CRYLSQ* of the *XRAY* system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). H atoms were located with *BONDAT* of the *XRAY* system and were kept fixed during the refinement. From the 385 parameters, 199 were refined; all non-H atoms were anisotropically refined; weighting scheme $w = 1/\{1 + [(F-B)/A]^2\}$ with $A = 30$ and $B = 60$; final $R = 0·083$, $wR = 0·071$, $S = 1·552$, $(\Delta/\sigma)_{\text{max}} = 0·16$, residual electron density from −0·69 to +0·71 e Å^{-3}; no secondary extinction; atomic scattering factors from *International Tables for X-ray Crystallography* (Ibers & Hamilton, 1974). The rather high final R value is probably caused by the high proportion of weak reflections in the measured range. The best-plane parameters given in the deposited data were calculated with the program *BP*70 (Ito & Sugawara, 1983).

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2) *for the non*-H *atoms in DCPOP*

	x	y	z	B_{eq}*
Cl(1)	0·7138 (5)	0·5500 (3)	0·1518 (2)	5·4
Cl(2)	0·9054 (5)	0·1816 (3)	−0·0051 (2)	6·0
N(1)	1·0057 (12)	0·2845 (7)	0·1575 (4)	3·5
O(1)	1·0159 (10)	0·1962 (6)	0·2056 (3)	4·0
O(2)	0·4854 (10)	0·1874 (7)	0·0557 (4)	4·8
C(1)	0·6956 (13)	0·1943 (8)	0·1534 (5)	2·7
C(2)	0·5530 (15)	0·1327 (9)	0·1038 (5)	3·5
C(3)	0·4766 (17)	0·0080 (10)	0·1173 (6)	4·9
C(4)	0·6202 (19)	−0·0934 (10)	0·1038 (6)	4·9
C(5)	0·7870 (17)	−0·1096 (9)	0·1582 (6)	5·0
C(6)	0·9257 (16)	−0·0009 (9)	0·1677 (6)	4·3
C(7)	0·8428 (13)	0·1159 (8)	0·1972 (5)	2·8
C(8)	0·8311 (14)	0·2850 (8)	0·1255 (5)	2·7
C(9)	0·5656 (14)	0·2550 (9)	0·2033 (5)	3·7
C(10)	0·6700 (17)	0·2302 (10)	0·2719 (6)	5·0
C(11)	0·7700 (17)	0·1068 (10)	0·2648 (5)	4·7
C(12)	0·8001 (14)	0·3666 (10)	0·0702 (5)	3·4
C(13)	0·7462 (14)	0·4924 (9)	0·0763 (6)	3·9
C(14)	0·7141 (17)	0·5684 (10)	0·0235 (8)	5·7
C(15)	0·7396 (16)	0·5287 (15)	−0·0384 (8)	5·7
C(16)	0·7965 (18)	0·4085 (14)	−0·0472 (6)	5·2
C(17)	0·8242 (14)	0·3317 (10)	0·0057 (6)	4·0

* $B_{\text{eq}} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Interatomic distances* (Å)

Cl(1)—C(13)	1·70 (1)	C(1)—C(9)	1·57 (1)
Cl(2)—C(17)	1·76 (1)	C(9)—C(10)	1·54 (2)
C(1)—C(2)	1·50 (1)	C(10)—C(11)	1·53 (2)
C(2)—C(3)	1·50 (2)	C(11)—C(7)	1·52 (1)
C(3)—C(4)	1·53 (2)	C(8)—C(12)	1·44 (1)
C(4)—C(5)	1·53 (2)	C(12)—C(13)	1·43 (1)
C(5)—C(6)	1·53 (2)	C(13)—C(14)	1·36 (2)
C(6)—C(7)	1·55 (1)	C(14)—C(15)	1·36 (2)
C(7)—C(1)	1·55 (1)	C(15)—C(16)	1·39 (2)
C(1)—C(8)	1·51 (1)	C(16)—C(17)	1·37 (2)
C(8)—N(1)	1·31 (1)	C(17)—C(12)	1·40 (2)
N(1)—O(1)	1·37 (1)	O(2)—C(2)	1·20 (1)
O(1)—C(7)	1·48 (1)		

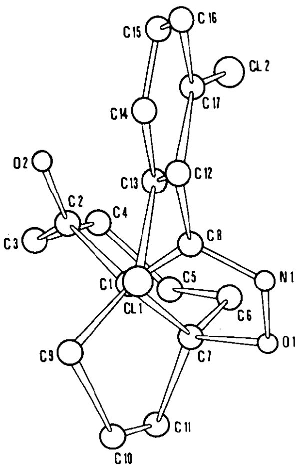


Fig. 1. Normal projection of the DCPOP molecule.

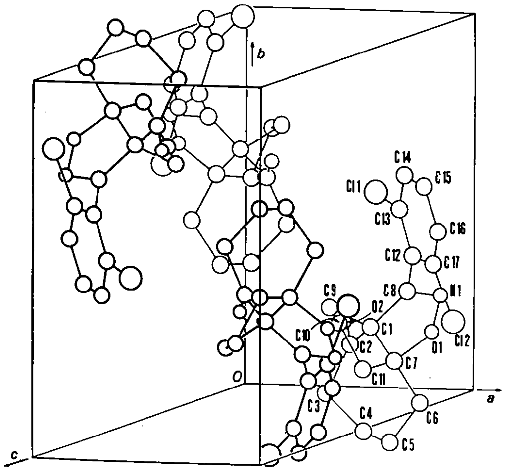


Fig. 2. Clinographic projection of the unit cell showing the molecular packing.

Discussion. The final positional parameters and equivalent isotropic temperature factors for the non-H atoms are given in Table 1.* Interatomic distances are in Table 2. A normal projection of the molecule with the atomic numbering is shown in Fig. 1, while a clinographic projection of the unit cell showing the molecular packing is given in Fig. 2. The two five-membered dihydroisoxazole and cyclopentane rings form an angle of 103·7 (4)°. The bonding of the two bridged C atoms C(1) and C(7) approximates to sp^3 hybridization. The dichlorophenyl ring is planar to within 0·04 Å. The two five-membered rings deviate considerably from planarity. The cycloheptanone ring has a chair conformation, which is energetically more stable than that of the boat alternative with torsion angles comparable to those in 2-bromodihydroisophoto-α-santonic lactone acetate (Asher & Sim, 1965). The mean plane of the cycloheptanone ring almost bisects the angle of the planes of the two five-membered rings, forming with them angles of 114·2 (4) and 138·4 (3)°. Owing to the C=O and C=N double bonds, the geometries around C(2) and C(8) are sp^2. The plane of the dichlorophenyl ring is almost perpendicular, 84·8 (3)°, to the plane of the dihydroisoxazole ring due to the strain caused by the interaction between the carbonyl group and the *o*-Cl-substituted phenyl ring. All bond lengths and angles are in good agreement with accepted values.

Thanks are due to Professor N. E. Alexandrou for useful suggestions and to the University of Thessaloniki for the use of its computing facilities.

---

* Lists of structure amplitudes, anisotropic thermal parameters, coordinates and isotropic temperature factors for H atoms, bond angles, least-squares-plane calculations and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43672 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Asher, J. D. M. & Sim, G. A. (1965). *J. Chem. Soc.* pp. 1584–1594.
Herr, M. L. (1977). *Tetrahedron,* **33**, 1897–1903.
Ibers, J. A. & Hamilton, W. C. (1974). *International Tables for X-ray Crystallography,* Vol. IV, pp. 71–151. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
Ito, T. C. & Sugawara, V. (1983). *Best Plane Program BP70.* Institute of Physical and Chemical Research, RIKEN, Wako, Japan.
Main, P. (1970). *DATRED. A Computer Program for the Reduction of Raw Diffractometer Data to FOBS.* Univ. of York, England.
Main, P., Lessinger, L., Woolfson, M. M., Germain, G. & Declercq, J.-P. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain-la-Neuve, Belgium.
Malamidou-Xenikaki, E., Coutouli-Argyropoulou, E. & Alexandrou, N. (1985). *Tetrahedron Lett.* **26**, 4105–4106.
Stewart, J. M., Kruger, G. J., Ammon, H. L., Dickinson, C. W. & Hall, S. R. (1972). The *XRAY72* system–version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.
Stewart, J. M., Machin, P. A., Dickinson, C. W., Ammon, H. L., Heck, H. & Flack, H. (1976). The *XRAY76* system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

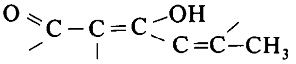
Acta Cryst. (1987). C**43**, 1153–1155

Structure of (5*RS*,6*RS*)-1,5-Diacetyl-6-benzoxymethyl-2-hydroxy-4-methyl-1,3-cyclohexadiene*

By E. Moreno, S. Perez, A. López-Castro and R. Márquez

Departamento de Optica y Sección de Física del Departamento de Investigaciones Físicas y Químicas del CSIC, Universidad de Sevilla, Spain

(*Received* 27 *May* 1986; *accepted* 12 *January* 1987)

Abstract. $C_{19}H_{20}O_5$, $M_r = 328·36$, triclinic, $P\bar{1}$, $a = 8·117$ (2), $b = 8·453$ (1), $c = 13·352$ (2) Å, $\alpha = 89·82$ (2), $\beta = 83·24$ (2), $\gamma = 113·38$ (2)°, $V = 833·8$ (3) Å^3, $Z = 2$, $D_m = 1·29$, $D_x = 1·30$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·108 mm^{-1}, $F(000) = 348$, $T = 293$ K, $R = 0·078$ for 2729 observed $[I > 2\sigma(I)]$ independent reflections. The cyclohexadiene ring conformation is twist–boat. There is some electron delocalization along the system  of the diacetylhydroxymethylcyclohexadiene group. The configuration around the chiral centres C(9) and C(11) of the cyclohexadiene ring is *R*,*R*. Packing of the molecules is governed by van der Waals contacts.

---

* [(1*RS*,6*RS*)-2,6-Diacetyl-3-hydroxy-5-methyl-2,4-cyclohexadienyl]methyl benzoate.

0108-2701/87/061153-03$01.50