anion with respect to the DMET stack is different from that of anions in other compounds. The $AuCl_2$ anion is located among eight DMET molecules.

Between the DMET columns, the Se—Se distance in $(DMET)_2AuCl_2$ is shorter than that in $(DMET)_2$-AuI_2. The contact mode is quite different in $(DMET)_2AuCl_2$ and $(DMET)_2AuI_2$; a comparison of the dimensionality is rather difficult. Similar electrical properties of the two compounds, however, suggest that the dimensionality is almost equal.

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Structure of 6-Chloro-10-oxabicyclo[5.2.1]decane-2,3-diol

BY M. L. RODRÍGUEZ, C. RUIZ-PÉREZ, L. DORTA, J. D. MARTÍN, E. ALVAREZ, E. MANTA AND D. ZURITA

Centro de Productos Naturales Orgánicos Antonio González, Universidad de La Laguna – CSIC, Carretera de la Esperanza 2, 38206 La Laguna, Tenerife, Spain

Abstract. $C_9H_{15}ClO_3$, $M_r = 206.7$, monoclinic, $P2_1/a$, $a = 11.627$ (2), $b = 7.486$ (4), $c = 11.625$ (5) Å, $\beta = 109.5$ (1)°, $V = 954$ (1) Å^3, $Z = 4$, $D_x = 1.44$ g cm^{-3}, $\lambda(Cu\ K\alpha) = 1.5418$ Å, $\mu = 33.8$ cm^{-1}, $F(000) = 440$, room temperature, $R = 0.074$ for 1123 reflections with $I \geq 2\sigma(I)$. The crystal structure is stabilized by extensive intermolecular hydrogen bonding, with a specific interaction between the hydroxyl (O3H) group of one molecule and the hydroxyl (O2H) group of another, forming hydrogen-bonded chains along the b axis. There is also an intramolecular hydrogen bond O2—H1···O3.

Introduction. Medium-size cyclic ethers are becoming increasingly recognized as common structural units in naturally occurring compounds, such as toxins (Shimizu, Chou, Bando, Duyne & Clardy, 1986; Murata, Kumagai, Soo Lee & Yasumoto, 1987) and marine natural products (Moore, 1978). Owing to the biological interest in these substances and the well recognized problems in building midsize rings, we are involved in the development of new synthetic methodologies for such molecules. Single-crystal X-ray analysis is a useful tool for solving structure-related problems. In this work we wish to report the molecular and crystal structure of the title compound synthesized in our laboratories (Alvarez, Manta, Martín, Rodríguez & Ruiz-Pérez, 1988; Alvarez, Manta, Martín, Rodríguez, Ruiz-Pérez & Zurita, 1988).

Table 1. *Final fractional atomic coordinates with their e.s.d.'s and equivalent isotropic thermal parameters (Å²) for non-H atoms*

$$B_{eq} = (1/3)\Sigma_i\Sigma_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cl	0·5417 (1)	0·1807 (2)	0·4051 (<1)	3·4 (1)
O1	0·5098 (3)	0·5795 (4)	0·1720 (3)	2·4 (1)
O2	0·7541 (3)	0·8331 (4)	0·1252 (3)	2·4 (1)
O3	0·8554 (3)	0·5155 (4)	0·0989 (3)	2·6 (1)
C1	0·6857 (4)	0·6722 (5)	0·1224 (4)	1·7 (2)
C2	0·7718 (4)	0·5147 (6)	0·1666 (4)	1·8 (2)
C3	0·7105 (6)	0·3342 (6)	0·1588 (5)	2·8 (2)
C4	0·6855 (5)	0·2657 (7)	0·2725 (5)	4·0 (3)
C5	0·5991 (4)	0·2989 (6)	0·2751 (4)	2·2 (2)
C6	0·5203 (5)	0·4897 (6)	0·2846 (4)	3·3 (2)
C7	0·6068 (9)	0·5995 (8)	0·3855 (6)	7·3 (4)
C8	0·6603 (5)	0·7365 (8)	0·3305 (4)	3·7 (2)
C9	0·6011 (4)	0·7170 (6)	0·1922 (4)	2·3 (2)

Table 2. *Bond lengths (Å) and angles (°) with their e.s.d.'s in parentheses*

Cl—C5	1·820 (2)	C2—C3	1·516 (1)
O1—C6	1·440 (1)	C3—C4	1·533 (1)
O1—C9	1·441 (1)	C4—C5	1·501 (1)
O2—C1	1·438 (1)	C5—C6	1·513 (1)
O3—C2	1·440 (1)	C6—C7	1·509 (1)
C1—C2	1·521 (1)	C7—C8	1·453 (1)
C1—C9	1·507 (1)	C8—C9	1·530 (1)
C6—O1—C9	110·5 (3)	C4—C5—C6	118·4 (4)
O1—C9—C1	109·3 (4)	Cl—C5—C4	108·5 (3)
C2—C1—C9	117·7 (4)	Cl—C5—C6	106·3 (3)
O2—C1—C9	104·9 (3)	O1—C6—C5	108·1 (4)
O2—C1—C2	110·2 (4)	C5—C6—C7	115·7 (5)
C1—C2—C3	115·3 (4)	O1—C6—C7	106·6 (4)
O3—C2—C1	107·5 (4)	C6—C7—C8	108·3 (5)
O3—C2—C3	111·1 (4)	C7—C8—C9	106·6 (5)
C2—C3—C4	117·9 (4)	C1—C9—C8	116·2 (4)
C3—C4—C5	115·5 (5)	O1—C9—C8	106·8 (4)

Experimental. A colourless crystal (0·5 × 0·4 × 0·4 mm) was selected and mounted on a computer-controlled four-circle Siemens AED diffractometer, Cu $K\alpha$ graphite-monochromated radiation. Unit-cell parameters from 20 reflections ($18 \le 2\theta \le 38°$), refined by least squares. 1241 independent reflections were measured in the range $3 \le 2\theta \le 110°$, hkl index: 0 to 12, 0 to 7, −12 to 12, max. $(\sin\theta)/\lambda = 0·5325$ Å^{-1}. Two standard reflections measured every hour showed no significant intensity decay. 1153 reflections (93%) with $I \ge 2\sigma(I)$ were taken as observed and used for structure determination after correction for Lorentz and polarization factors.

The Cl atom was located from a Patterson synthesis (Sheldrick, 1986) and the remaining non-H atoms from Fourier syntheses (Sheldrick, 1976). In the course of the isotropic refinement of the positional parameters of the non-H atoms, an empirical absorption correction was performed with the *DIFABS* program (Walker & Stuart, 1983). The minimum and maximum corrections were 0·682 and 1·314. H atoms located from difference Fourier syn-

thesis maps. Anisotropic full-matrix least-squares refinement on F, non-H atoms anisotropic, some H atoms included using a riding model with C—H 1·08 Å, and the remaining refined freely with the program *SHELX*76 (Sheldrick, 1976). Final $R = 0·074$, $wR = 0·087$ for 1123 observed reflections and 144 parameters. The weighting scheme was $w^{-1} = \sigma^2(F) + 0·000001F^2$. Max. shift/e.s.d. 0·2, max. features in final difference map $+0·24$, $-0·21$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).
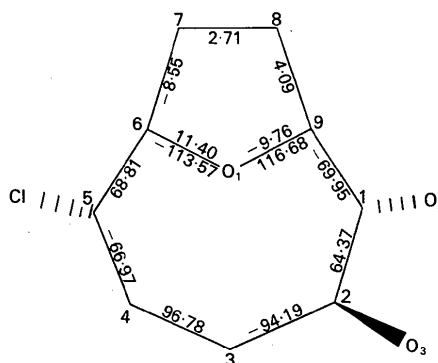

Fig. 1. Selected torsion angles (°, e.s.d.'s ~0·5°) and molecular scheme for the title compound.
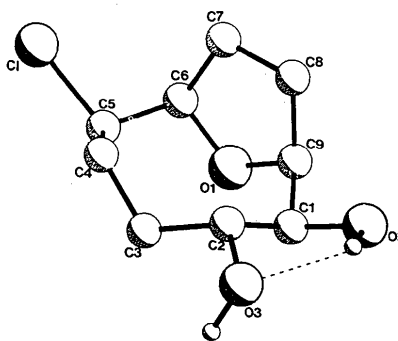

Fig. 2. View of $C_9H_{15}ClO_3$, showing the atomic numbering (only H atoms of the hydroxyl groups included). Intramolecular hydrogen bonding is shown.
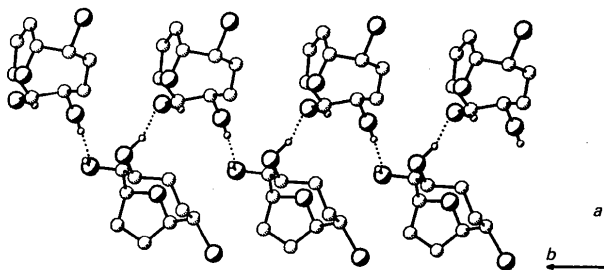

Fig. 3. Hydrogen-bonding scheme of the title compound.

Discussion. Table 1 lists the fractional atomic coordinates of the non-H atoms,* Table 2 the bond lengths and angles. Fig. 1 shows selected torsion angles.

The structure of the title compound is illustrated in Fig. 2, which includes the atomic numbering scheme. As indicated by the torsion angles (Fig. 1) and bond distances (Table 2), the eight-membered ring shows approximate m symmetry with the pseudoplane including the C3–O1 vector. The five-membered ring has an approximate twofold rotation axis bisecting the O1—C6 bond and the C8 atom. The molecule shows intramolecular hydrogen bonding: O2—H1···O3 2·715 (5) Å (Fig. 2). Fig. 3 gives a representation of the hydrogen-bonding scheme; the donor–acceptor distance is 2·839 (5) Å. The crystal structure is stabilized by extensive hydrogen bonding, with a specific interaction between the hydroxyl

group O3H of one molecule and O2H of another, forming hydrogen-bonded chains along the b axis.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52277 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of Substituted Olefins. I. (Z)-2-Nitro-1,2-diphenylvinyl Thiocyanate

By KYONG PAE PARK AND JONG HWA JEONG

Division of Chemistry, Korea Institute of Science and Technology, Cheongryang, PO Box 131, Seoul 136-791, Korea

Abstract. $C_{15}H_{10}N_2O_2S$, $M_r = 282\cdot3$, orthorhombic, $Pbca$, $a = 15\cdot467$ (3), $b = 16\cdot251$ (4), $c = 11\cdot235$ (1) Å, $V = 2824\cdot0$ (9) Å^3, $Z = 8$, $D_x = 1\cdot328$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71073$ Å, $\mu = 1\cdot87$ cm^{-1}, $F(000) = 1168$, $T = 298$ K, $R = 0\cdot040$ for 1394 unique reflections with $I > 3\sigma(I)$. The C atoms of the C=C double bond, the N and O atoms of nitro group, and the S atom of the thiocyanate group form a nearly planar backbone. The two phenyl groups are nearly perpendicular to the backbone of the molecule.

Introduction. The configuration of olefinic compounds can be determined by various methods such as ^{1}H NMR, IR and dipole-moment measurements. These methods are occasionally not sufficient to give all structural information, while X-ray structure analysis can provide the absolute structure. (Z)-2-Nitro-1,2-diphenylvinyl thiocyanate is one of a series of multiply substituted olefins investigated for biological activity. Its configuration has significant implications in nucleophilic vinylic substitution. Its published *trans*-diphenyl configuration (Rapport & Topol, 1980) is apparently in error. These two aspects will be presented in detail elsewhere.

Experimental. A pale-yellow single crystal having dimensions $0\cdot25 \times 0\cdot33 \times 0\cdot35$ mm was sealed in a thin-walled glass capillary. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation [$\lambda(K\alpha_1) = 0\cdot70930$, $\lambda(K\alpha_2) = 0\cdot71359$ Å], was used for collecting intensity data. Unit-cell parameters were determined by least-squares refinement of 25 reflections ($10 \le \theta \le 16°$). Data collection was conducted at room temperature in ω–2θ scan mode using variable rate, $\theta_{\max} = 25°$ for the range $0 \le h \le 18$, $0 \le k \le 19$, $0 \le l \le 13$, three reflections monitored every hour over the course of data collection for which a linear correction (maximum correction factor 1·004) was applied for 1582 intensities.