

## References

- ACHESON, R. M., FERRIS, M. J., CRITCHLEY, S. R. & WATKIN, D. J. (1980). *J. Chem. Soc. Perkin Trans. 2*, pp. 326–329.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- CULVENOR, C. C. J., EDGAR, J. A., SMITH, L. W. & TWEEDDALE, H. J. (1970). *Aust. J. Chem.* **23**, 1853–1867.
- CULVENOR, C. C. J. & WOODS, W. G. (1965). *Aust. J. Chem.* **18**, 1625–1637.
- ENGLISH, R. B., MCGILLIVRAY, G. & SMAL, E. (1980). *Acta Cryst.* **B36**, 1136–1141.
- HUXTABLE, R. J. (1979). *Gen. Pharmacol.* **10**, 159–167.
- HUXTABLE, R. J. (1980). *Trends Pharmacol. Sci.* **1**, 299–303.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- MACKAY, M. F. & CULVENOR, C. C. J. (1982). *Acta Cryst.* **B38**, 2754–2758.
- MACKAY, M. F., SADEK, M. & CULVENOR, C. C. J. (1984). *Acta Cryst.* **C40**, 470–472.
- MACKAY, M. F., SADEK, M., CULVENOR, C. C. J. & SMITH, L. W. (1983). *Acta Cryst.* **C39**, 1230–1233.
- MCLEAN, E. K. (1970). *Pharmacol. Rev.* **22**, 429–483.
- SHELDRIK, G. M. (1976). *SHELX76*. A program for crystal structure determination. Univ. of Cambridge, England.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- STOECKLI-EVANS, H. (1979). *Acta Cryst.* **B35**, 231–234.
- STOECKLI-EVANS, H. & ROBINS, D. T. (1983). *Helv. Chim. Acta*, **66**, 1376–1380.
- SUSSMAN, J. L. & WODAK, S. J. (1973). *Acta Cryst.* **B29**, 2918–2926.
- TASHKHODZHAEV, B., YAGUDAEV, M. R. & YUNUSOV, S. YU. (1979). *Khim. Priir. Soedin.* **3**, 368–373.
- WANG, S. (1981). *Sci. Sin.* **24**, 497–507.

*Acta Cryst.* (1984). **C40**, 476–479

## Chiral and Hexacoordinated Chloro(methyl)bis(8-quinolinolato)silicon(IV), Si(CH<sub>3</sub>)(C<sub>9</sub>H<sub>6</sub>NO)<sub>2</sub>Cl\*

BY GERHARD KLEBE† AND DUC TRAN QUI

*Laboratoire de Cristallographie, Centre National de la Recherche Scientifique, 25 avenue des Martyrs, F-3800 Grenoble, France*

(Received 21 April 1983; accepted 13 October 1983)

**Abstract.**  $M_r = 366.89$ , triclinic,  $P\bar{1}$ ,  $a = 8.085$  (2),  $b = 8.195$  (1),  $c = 15.040$  (5) Å,  $\alpha = 76.14$  (2),  $\beta = 74.59$  (5),  $\gamma = 60.41$  (2)°,  $V = 828.3$  (3) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.465$  Mg m<sup>-3</sup>, Mo  $K\alpha$  (graphite monochromator),  $\lambda = 0.71069$  Å,  $\mu = 0.320$  mm<sup>-1</sup>,  $F(000) = 380$ ,  $T = 295$  K,  $R_w(F) = 0.049$  using 2922 independent reflections. Hexacoordinated silicon is found in a distorted octahedral geometry. Hexacoordination is achieved by intramolecular Lewis acid–base interaction. The coordinative Si–N distance is 2.015 (4) Å, the Si–O bond length 1.765 (2) Å. The bidentate ligands are arranged in *cis* configuration along two edges of the octahedral polyhedron and methyl and chlorine are found at the remaining sites in equipopulation. Si represents a center of chirality and two isomeric forms are present in the crystal, which correspond to the left- and right-hand arrangements.

**Introduction.** Although silicon and carbon have the same number of valence electrons, there are pronounced differences in the chemical behavior of the two

elements. Many of these dissimilarities are attributed to an expansion of the valence shell of silicon, which leads to compounds and suggested reaction intermediates with coordination number higher than four. Penta- and hexavalency have been detected in neutral and charged complexes when the central atom is bonded to electronegative elements like O, N or F, e.g. Si(C<sub>11</sub>H<sub>12</sub>N<sub>2</sub>)Cl<sub>3</sub> (Klebe, Bats & Hensen, 1983a), SiF<sub>4</sub>(pyridine)<sub>2</sub> (Bain, Killeen & Webster, 1969), SiF<sub>6</sub><sup>2-</sup> (Frlec, Gantar, Golič & Leban, 1980).

**Experimental.** The compound was prepared from lithium quinolinolate, (C<sub>9</sub>H<sub>6</sub>NO)Li, and CH<sub>3</sub>SiCl<sub>3</sub> as described by Hensen & Klebe (1981): pale yellow crystals from slow evaporation of a saturated solution in CH<sub>2</sub>Cl<sub>2</sub>; pronounced sensitivity to moisture, so crystals selected under dried argon and sealed in thin glass tubes; crystal 0.3 × 0.2 × 0.2 mm; space group determined by film methods to be  $P1$  or  $P\bar{1}$ ; lattice parameters refined from 25 reflections on a Nonius CAD-4 diffractometer.  $\sin\theta/\lambda \leq 0.594$  Å<sup>-1</sup>,  $\omega$ -scan mode, scan width: (1.4 + 0.35 tan $\theta$ )°, 5380 reflections,  $h - 9-5$ ,  $k - 9-9$ ,  $l - 17-17$ , three standard reflections, no significant fluctuations, no correction for absorption; background correction and averaging, 2922 unique reflections [ $R_{\text{int}} = 0.044$ , 641 unobserved,  $I < \sigma(I)$ ] for

\* Coordinative Interactions in Chelated Complexes of Silicon. Part X. Part IX: Klebe, Nix & Hensen (1983).

† Present address: Institut für Kristallographie und Mineralogie der Universität Frankfurt/Main, Senckenberganlage 30, D-6000 Frankfurt/Main 1, Federal Republic of Germany.

structure determination. Part of the octahedral coordination around silicon found by direct methods (*MULTAN*: Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); additional atoms detected by difference Fourier, refined on *F* with XRAY (Stewart, 1975) in centric space group,  $R_w(F) = 0.049$ ,  $R(F) = 0.087$ , anisotropic temperature factors for non-hydrogen atoms [ $w = 1/\sigma^2(F)$ ], H atoms included in positional refinement with fixed overall temperature factor. Disorder was discovered between a chlorine and a methyl carbon. Positions at the corners of one edge of the octahedron around Si were split into two scattering centers partly occupied by Cl and C; refinement of population parameters revealed an equipopulation in the two sites; atomic form factors for H from Stewart, Davidson & Simpson (1965), for other atoms from *International Tables for X-ray Crystallography* (1974).

**Discussion.** Final atomic positions are given in Table 1 and the bond lengths and angles in Table 2.\*

Silicon is at the center of a distorted octahedron (Fig. 1). The two bidentate ligands are arranged nearly perpendicular [ $86.0(3)^\circ$ ] to each other in a *cis* geometry and Si therefore represents a center of chirality. The *cis* topology is in agreement with several examples of hexacoordinated complexes with bidentate ligands like *o*-phenanthroline, bipyridine or 8-quinoline (Sawitzki, von Schnering, Kummer & Seshadri, 1978; Bellavance, Corey, Corey & Hey, 1977). It can be explained from stereochemical reasons: the *cis*

\* Lists of structure factors, bond angles and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38914 (35 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

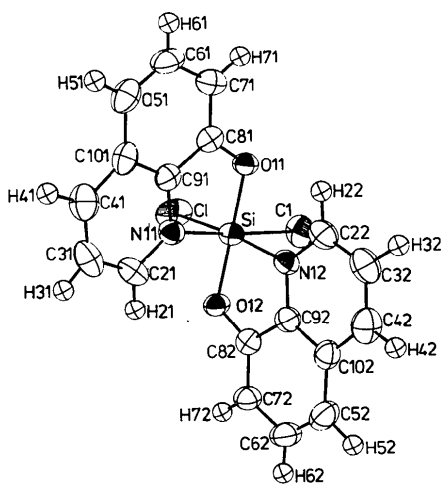


Fig. 1. ORTEP drawing of the complex; thermal ellipsoids are drawn at 50% probability level; H atoms, shown as spheres, have arbitrary radius.

Table 1. Final fractional coordinates and the arithmetic mean of the thermal ellipsoids projected on the principal axis, *e.s.d.*'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}(\text{\AA}^2)$
C(1)	1.188 (5)	0.098 (4)	0.692 (2)	0.05 (1)†
C(1')	0.975 (4)	0.394 (3)	0.806 (2)	0.009 (5)†
Cl	1.224 (1)	0.0782 (9)	0.6858 (5)	0.034 (2)†
Cl'	0.989 (1)	0.425 (1)	0.815 (6)	0.033 (2)†
Si	0.9245 (1)	0.2510 (1)	0.7501 (7)	0.0276 (5)
O(11)	0.9416 (3)	0.0763 (3)	0.8471 (1)	0.034 (1)
O(12)	0.8649 (3)	0.4254 (3)	0.6525 (1)	0.026 (1)
N(11)	0.6458 (4)	0.3801 (3)	0.8114 (2)	0.029 (1)
N(12)	0.8370 (4)	0.1223 (4)	0.6875 (2)	0.029 (1)
C(21)	0.4994 (5)	0.5407 (5)	0.7869 (2)	0.036 (2)
C(22)	0.8263 (5)	-0.0398 (5)	0.7123 (3)	0.035 (2)
C(31)	0.3186 (5)	0.6107 (5)	0.8464 (3)	0.041 (2)
C(32)	0.7749 (5)	-0.1086 (5)	0.6529 (3)	0.041 (2)
C(41)	0.2923 (5)	0.5114 (5)	0.9309 (3)	0.041 (2)
C(42)	0.7348 (5)	-0.0102 (5)	0.5675 (3)	0.037 (2)
C(51)	0.4441 (5)	0.2254 (5)	1.0470 (2)	0.036 (2)
C(52)	0.7166 (5)	0.2762 (5)	0.4515 (3)	0.034 (2)
C(61)	0.6076 (6)	0.0661 (5)	1.0650 (2)	0.035 (2)
C(62)	0.7386 (5)	0.4344 (5)	0.4339 (2)	0.042 (2)
C(71)	0.7817 (5)	0.0062 (4)	0.9995 (2)	0.030 (2)
C(72)	0.7869 (5)	0.4970 (4)	0.5000 (2)	0.035 (2)
C(81)	0.7874 (5)	0.1133 (4)	0.9154 (2)	0.026 (2)
C(82)	0.8157 (4)	0.3883 (5)	0.5832 (2)	0.027 (2)
C(91)	0.6201 (5)	0.2815 (4)	0.8958 (2)	0.025 (2)
C(92)	0.7977 (4)	0.2199 (4)	0.6024 (2)	0.030 (2)
C(101)	0.4448 (5)	0.3420 (5)	0.9600 (2)	0.030 (2)
C(102)	0.7466 (4)	0.1605 (5)	0.5390 (2)	0.033 (2)
H(21)	0.539 (4)	0.608 (4)	0.727 (2)	
H(22)	0.867 (4)	-0.115 (4)	0.774 (2)	
H(31)	0.207 (4)	0.738 (4)	0.832 (2)	
H(32)	0.778 (4)	-0.224 (4)	0.678 (2)	
H(41)	0.156 (4)	0.560 (4)	0.968 (2)	
H(42)	0.701 (4)	-0.071 (4)	0.525 (2)	
H(51)	0.326 (4)	0.263 (4)	1.098 (2)	
H(52)	0.689 (4)	0.234 (4)	0.407 (2)	
H(61)	0.612 (4)	-0.013 (4)	1.122 (2)	
H(62)	0.721 (4)	0.508 (4)	0.380 (2)	
H(71)	0.886 (4)	-0.105 (4)	1.012 (2)	
H(72)	0.796 (4)	0.615 (4)	0.486 (2)	

† Half populated.

Table 2. Bond lengths (Å) and selected angles (°)

Si—C(1)	1.94 (3)	C(31)—C(41)	1.362 (5)
Si—C(1')	1.83 (3)	C(32)—C(42)	1.368 (6)
Si—Cl	2.199 (8)	C(41)—C(101)	1.395 (4)
Si—Cl'	2.217 (12)	C(42)—C(102)	1.405 (6)
Si—O(11)	1.763 (2)	C(51)—C(61)	1.357 (5)
Si—O(12)	1.766 (2)	C(51)—C(101)	1.424 (5)
Si—N(11)	2.014 (3)	C(52)—C(62)	1.349 (7)
Si—N(12)	2.016 (4)	C(52)—C(102)	1.426 (5)
O(11)—C(81)	1.341 (4)	C(61)—C(71)	1.416 (5)
O(12)—C(82)	1.352 (5)	C(62)—C(72)	1.432 (7)
N(11)—C(21)	1.316 (4)	C(71)—C(81)	1.360 (4)
N(11)—C(91)	1.357 (4)	C(72)—C(82)	1.352 (4)
N(12)—C(22)	1.331 (5)	C(81)—C(91)	1.412 (4)
N(12)—C(92)	1.362 (4)	C(82)—C(92)	1.412 (6)
C(21)—C(31)	1.411 (5)	C(91)—C(101)	1.412 (4)
C(22)—C(32)	1.398 (7)	C(92)—C(102)	1.395 (6)
C(1)—Si—C(1')	98 (2)		
C(1)—Si—Cl	97 (1), 96.9 (8)		
C(1)—Si—O(1)	92.2 (8), 95.0 (8), 95.2 (6), 92.4 (6)		
C(1)—Si—N(1)	172 (1), 88 (1), 88.8 (7), 172.9 (6)		
Cl—Si—Cl'	96.1 (4)		
Cl—Si—O(1)	91.7 (2), 95.6 (2), 96.0 (2), 91.7 (2)		
Cl—Si—N(1)	172.9 (3), 89.2 (3), 89.7 (2), 173.0 (2)		
O(1)—Si—N(1)	83.6 (1), 88.2 (1), 88.3 (1), 83.4 (1)		
Si—O(1)—C(8)	117.8 (2), 117.9 (2)		
Si—N(1)—C(2)	131.7 (2), 131.5 (3)		
Si—N(1)—C(9)	109.4 (2), 109.9 (3)		
O(1)—C(8)—C(7)	125.9 (2), 125.1 (4)		

geometry of the ligand minimizes the sterical interference between the hydrogen atoms in 2 and 7 positions of different ring systems. The impact of this interaction on the molecular build up is shown in crystal structures of hexacoordinated complexes with, for example, four pyridine ligands in the base plane that represent a propeller-like arrangement of the aromatic cycles (Sinclair, Small & Worrall, 1981; Pullmann, Hensen & Bats, 1982). In the title compound the chelate ligands are connected to the central atom through a Si—O 'single bond' and a Si—N 'coordinative bond'. The two Si—O bonds form an almost linear arrangement [168.7(1)°]. The N—Si—N angle is 85.3(1)°. At the sites across from the nitrogens, C-methyl and chlorine are observed in equipopulation. The Si—Cl and Si—C bonds located across from the nitrogens represent elongated values compared to those in tetravalent derivatives.

The C/Cl disorder displays the presence of two geometrical isomeric forms in the crystal. Each of them is transformed into its enantiomer by the inversion center of  $P\bar{1}$ . Starting with one molecular arrangement the C/Cl exchange leads to the other isomeric geometry which would only be identical with the first one and its mirror image if the arrangement around Si, including the four centers involved in the disorder, possessed exact  $C_2$  point symmetry. This restriction is not fulfilled in the present case. The distribution of the two isomers might be ordered in  $P1$  with the two forms, which are close to enantiomeric to each other, as symmetry-independent molecules in the unit cell or statistical in  $P\bar{1}$  (as presumed in the structure refinement).

The two Si—O bonds are significantly longer than the 1.64(3) Å observed as a mean value in tetracoordinated siloxanes (Voronkov, Mieshkevich & Yuzhelevskii, 1978). They are much more extended than the Si—O bonds (mean: 1.66 Å) in the equatorial plane of pentacoordinated silatranes (Boer, Turley & Flynn, 1968; Turley & Boer, 1968, 1969; Párkányi, Simon & Nagy, 1974, 1975). In the present complex the siloxane bonds are comparable with those in penta- and hexavalent silicate anions (Table 3), but they are shorter than the shortest Si...O coordination bond of 1.918(4) Å (Onan, McPhail, Yoder & Hillyard, 1978).

The two Si...N bonds are remarkably extended in comparison to 'normal' Si—N 'single bonds' (mean: 1.75 Å). The expansion (13% based on 1.75 Å as 'normal' Si—N single bond) is in the same range as in other halogen-containing penta- and hexacoordinated silicon complexes (Klebe, Bats & Hensen, 1983*b*). The bond angles around silicon to atoms on facing sites of the octahedron are all close to 170°. The perturbation from linear arrangements might occur from the severe strain in the five-membered ring. The angles O(1)—C(8)—C(9) [114.9(3)°] and C(8)—C(9)—N(1) [113.6(3)°] are reduced substantially below the normal

Table 3. Si—O distances in four penta- and hexacoordinated silicate structures (Å)

Bis(1,2-benzenediolato)-(phenyl)silicate <sup>a</sup>	1.700 (9) <sub>eq</sub> 1.794 (5) <sub>ax</sub>
Bis(1,2-benzenediolato)-fluorosilicate <sup>b</sup>	1.742 (5) 1.741 (5) 1.736 (5) 1.738 (5) 1.704 (5) 1.699 (5) 1.706 (5) 1.701 (5)
Bis(1,2-ethanediolato)-(methyl)silicate <sup>c</sup>	1.769 (2) 1.711 (2) 1.749 (2) 1.735 (2)
Tris(1,2-benzenediolato)-silicate <sup>d</sup>	1.813 (3) 1.755 (3) 1.765 (3)

References: (a) Boer, Flynn & Turley (1968); (b) Harland, Day, Vallano, Sau & Holmes (1981); (c) Schomburg (1982); (d) Flynn & Boer (1969).

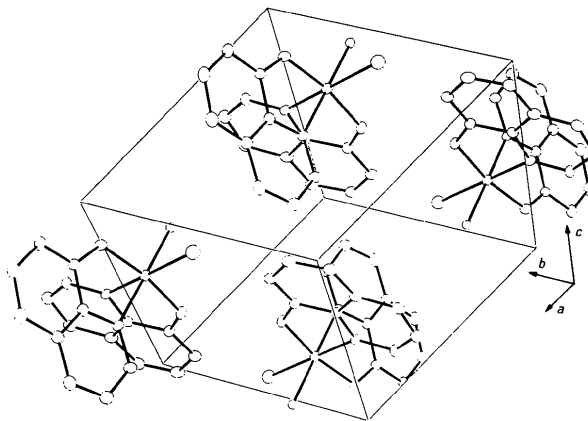


Fig. 2. The packing arrangement within a unit cell.

value of 120° found at 'sp<sup>2</sup>'-hybridized carbon or nitrogen. The angles around silicon [83.4(1), 83.6(1)°] at the vertex of the five-membered rings are smaller than in ideal octahedral geometry and in the above-mentioned silicates (Table 3). The reason for this deviation is probably the substitution of a Si—O bond (Table 3) by a much longer Si—N coordination bond, thereby allowing a smaller angle at silicon. The C—O bond [C(8)—O(1)] to the quinoline system is shorter than the 'normal' single-bond length in ethers and alcohols (> 1.4 Å), but it aligns with the C—O distance in 8-quinolinolium chloride (Klebe & Tran Qui, 1983). Chemically equivalent but crystallographically independent bonds in the two ligand skeletons resemble each other quite well. The bond angles within the aromatic ring systems are found between 115.9(3) and 124.0(3)°. The molecular packing in the triclinic unit cell is shown in Fig. 2.

#### References

- BAIN, V. A., KILLEAN, R. C. G. & WEBSTER, M. (1969). *Acta Cryst.* **B25**, 156–159.  
 BELLAVANCE, P. L., COREY, E. R., COREY, J. Y. & HEY, G. W. (1977). *Inorg. Chem.* **16**, 462–467.

- BOER, F. P., FLYNN, J. J. & TURLEY, J. W. (1968). *J. Am. Chem. Soc.* **90**, 6973–6977.
- BOER, F. P., TURLEY, J. W. & FLYNN, J. J. (1968). *J. Am. Chem. Soc.* **90**, 5102–5105.
- FLYNN, J. J. & BOER, F. P. (1969). *J. Am. Chem. Soc.* **91**, 5756–5761.
- FRLEC, B., GANTAR, D., GOLIC, L. & LEBAN, I. (1980). *Acta Cryst.* **B36**, 1917–1918.
- HARLAND, J. J., DAY, R. O., VALLANO, J. F., SAU, A. C. & HOLMES, R. R. (1981). *J. Am. Chem. Soc.* **103**, 5269–5270.
- HENSEN, K. & KLEBE, G. (1981). *J. Organomet. Chem.* **209**, 17–23.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.
- KLEBE, G., BATS, J. W. & HENSEN, K. (1983a). *Z. Naturforsch. Teil B*, **38**, 825–829.
- KLEBE, G., BATS, J. W. & HENSEN, K. (1983b). Submitted to *J. Chem. Soc. Dalton Trans.*
- KLEBE, G., NIX, M. & HENSEN, K. (1983). *Chem. Ber.* In the press.
- KLEBE, G. & TRAN QUI, D. (1983). In preparation.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- ONAN, K. D., MCPHAIL, A. T., YODER, C. H. & HILLYARD, R. W. (1978). *J. Chem. Soc. Chem. Commun.* pp. 209–210.
- PÁRKÁNYI, L., SIMON, K. & NAGY, J. (1974). *Acta Cryst.* **B30**, 2328–2332.
- PÁRKÁNYI, L., SIMON, K. & NAGY, J. (1975). *J. Organomet. Chem.* **101**, 11–18.
- PULLMANN, P., HENSEN, K. & BATS, J. W. (1982). *Z. Naturforsch. Teil B*, **37**, 1312–1315.
- SAWITZKI, G., VON SCHNERING, H. G., KUMMER, D. & SESHADRI, T. (1978). *Chem. Ber.* **111**, 3705–3710.
- SCHOMBURG, D. (1982). *Z. Naturforsch. Teil B*, **37**, 195–197.
- SINCLAIR, I., SMALL, R. W. H. & WORRALL, I. J. (1981). *Acta Cryst.* **B37**, 1290–1292.
- STEWART, J. M. (1975). Editor, XRAY system—version of June 1974, update January 1975. Computer Science Center, Univ. of Maryland, College Park, Maryland.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- TURLEY, J. W. & BOER, F. P. (1968). *J. Am. Chem. Soc.* **90**, 4026–4030.
- TURLEY, J. W. & BOER, F. P. (1969). *J. Am. Chem. Soc.* **91**, 4129–4134, 4134–4139.
- VORONKOV, M. G., MIESHKVICH, V. P. & YUZHELEVSKII, YU. A. (1978). *The Siloxane Bond*. New York: Consultants Bureau.

*Acta Cryst.* (1984). **C40**, 479–481

## Structure of 1-Hydroxy-5,11,15-trimethylpentacyclo[9.5.1.0<sup>2,9</sup>.0<sup>4,8</sup>.0<sup>12,16</sup>]heptadec-2(9)-en-3-one, C<sub>20</sub>H<sub>28</sub>O<sub>2</sub>\*

BY M. SORIANO-GARCIA,† B. ORTIZ AND R. A. TOSCANO

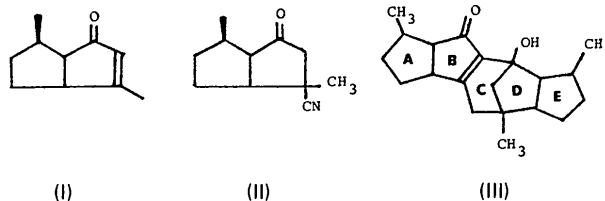
*Instituto de Química, UNAM, Circuito Exterior, Deleg. Coyoacán, 04510 México DF*

(Received 27 April 1983; accepted 21 October 1983)

**Abstract.**  $M_r = 300.4$ , orthorhombic,  $P2_12_12_1$ ,  $a = 10.677$  (4),  $b = 7.205$  (2),  $c = 22.749$  (4) Å,  $V = 1750$  Å<sup>3</sup>,  $D_x = 1.13$  Mg m<sup>-3</sup>,  $Z = 4$ ,  $F(000) = 656$ ,  $\text{Cu K}\alpha$ ,  $\lambda = 1.54178$  Å,  $\mu = 0.522$  mm<sup>-1</sup>,  $T = 300$  K,  $R = 0.050$  for 1095 observed reflections. The molecular structure is determined from the X-ray data and confirms the structure previously assigned on the basis of chemical and spectroscopic evidence. The molecule is stabilized by the formation of an intramolecular hydrogen bond between the hydroxyl and carbonyl groups [H(O1)⋯O(2) 2.047 (4) Å, O(1)–H(O1)⋯O(2) 152.8 (2)°].

**Introduction.** In the course of synthetic studies directed towards  $\alpha$ -piztol (Walls, Padilla, Joseph-Nathan, Giral, Escobar & Romo, 1966) we obtained an intermediate (III) whose structure required elucidation.

The ketonitrile (II) was obtained from the enone (I) (Achmad & Cavill, 1965; Setzer, Ortiz & Walls, 1980) by conjugated addition of HCN when treated with KCN in aqueous ethanol in the presence of ammonium chloride (Rodig & Johnston, 1969). Treatment of ketonitrile (II) with sodium hydride in refluxing benzene afforded (I) and the pentacyclic (III).



The X-ray crystallographic structural determination of (III) was undertaken in order to establish the crystal and molecular structure and the stereochemistry of this intermediate.

\* Contribution No. 648 of the Instituto de Química, UNAM.

† To whom correspondence should be addressed.