N(12)—C(13)—C(14)	114.6 (13)	N(1)—C(14)—C(5)	125.2 (17)
N(1)—C(14)—C(13)	119.6 (16)	C(5)—C(14)—C(13)	115.2 (15)
C(16)—N(15)—C(28)	119.2 (16)	N(15)—C(16)—C(17)	123.3 (15)
C(16)—C(17)—C(18)	117.5 (18)	C(17)—C(18)—C(19)	120 (2)
C(18)—C(19)—C(20)	119.3 (17)	C(18)—C(19)—C(28)	119.6 (15)
C(20)—C(19)—C(28)	121.0 (17)	C(19)—C(20)—C(21)	120.3 (17)
C(20)—C(21)—C(22)	122.0 (14)	C(21)—C(22)—C(23)	126.0 (15)
C(21)—C(22)—C(27)	118.0 (16)	C(23)—C(22)—C(27)	115.9 (17)
C(22)—C(23)—C(24)	119.3 (16)	C(23)—C(24)—C(25)	120.0 (19)
C(24)—C(25)—N(26)	122.7 (19)	C(25)—N(26)—C(27)	118.5 (13)
C(22)—C(27)—N(26)	123.5 (17)	C(22)—C(27)—C(28)	119.7 (16)
N(26)—C(27)—C(28)	116.8 (14)	N(15)—C(28)—C(19)	119.9 (17)
N(15)—C(28)—C(27)	121.2 (17)	C(19)—C(28)—C(27)	118.9 (15)
C(30)—N(29)—C(42)	116.2 (16)	N(29)—C(30)—C(31)	121.1 (17)
C(30)—C(31)—C(32)	119 (2)	C(31)—C(32)—C(33)	124 (2)
C(32)—C(33)—C(34)	127 (2)	C(32)—C(33)—C(42)	114.4 (18)
C(34)—C(33)—C(42)	119 (2)	C(33)—C(34)—C(35)	119.6 (19)
C(34)—C(35)—C(36)	124.8 (18)	C(35)—C(36)—C(37)	125.5 (17)
C(35)—C(36)—C(41)	118 (2)	C(37)—C(36)—C(41)	116.7 (18)
C(36)—C(37)—C(38)	121.8 (17)	C(37)—C(38)—C(39)	118 (2)
C(38)—C(39)—N(40)	122.6 (17)	C(39)—N(40)—C(41)	118.8 (13)
C(36)—C(41)—N(40)	121.7 (18)	C(36)—C(41)—C(42)	119.0 (17)
N(40)—C(41)—C(42)	119.1 (14)	N(29)—C(42)—C(33)	125.5 (19)
N(29)—C(42)—C(41)	114.8 (17)	C(33)—C(42)—C(41)	119.7 (16)

Table 3. *Average geometric values of the pseudo-equivalent bonds* (Å, °)

N(1)—C(2)	1.315 (11)	N(1)—C(14)	1.345 (9)
C(2)—C(3)	1.412 (13)	C(3)—C(4)	1.352 (13)
C(4)—C(5)	1.388 (13)	C(5)—C(14)	1.426 (11)
C(5)—C(6)	1.421 (11)	C(14)—C(13)	1.462 (16)
C(6)—C(7)	1.35 (2)		
C(2)—N(1)—C(14)	117.5 (6)	N(1)—C(2)—C(3)	123.0 (7)
C(2)—C(3)—C(4)	118.8 (8)	C(3)—C(4)—C(5)	121.1 (8)
C(4)—C(5)—C(14)	115.6 (7)	C(4)—C(5)—C(6)	125.5 (7)
C(14)—C(5)—C(6)	120.3 (8)	N(1)—C(14)—C(5)	123.9 (7)
N(1)—C(14)—C(13)	117.7 (6)	C(5)—C(14)—C(13)	118.4 (7)
C(5)—C(6)—C(7)	121.2 (7)		

The ω scan width was $(0.85 + 0.35\tan\theta)^\circ$ and the scan speed was 5.45–29.30° min^{-1}. The structure was solved by direct methods and difference Fourier synthesis, refined with unit weights by full-matrix least-squares methods with anisotropic displacement parameters for all non-H atoms.

Data collection: Siemens *R3m/V* software. *PLUTO* (Motherwell & Clegg, 1978) and all calculations were performed on a VMX computer with the *SHELXTL-Plus* (VMS) (Sheldrick, 1987) program package.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: TA1000). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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An $As_2Si_3O_6$ Cage Compound, 3,3,7,7,10,10-Hexaphenyl-2,4,6,8,9,11-hexaoxa-1,5-diarsa-3,7,10-trisilabicyclo[3.3.3]undecane

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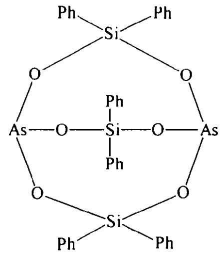
Abstract

The title compound, $C_{36}H_{30}As_2O_6Si_3$, is the first arsenosilicate compound containing tricoordinate arsenic(III) to be studied using X-ray crystallography. This is also the first structural determination of an arsenosilicate compound with the siloxy moiety acting as a bridging group. Two trigonal pyramidal AsO_3 units are joined by three tetrahedral —$OSiPh_2O$— bridges to form an 11-atom $As_2Si_3O_6$ cage with mean dimensions As—O 1.760 (15), Si—O 1.637 (5) Å, O—As—O 98.9 (10), O—Si—O 111.1 (4), As—O—Si 139.1 (13)°.

Comment

A small number of compounds containing terminal As—O—$SiMe_3$ units have been reported recently (Baier, Bissinger & Schmidbaur, 1992, 1993; Baier, Paul & Schmidbaur, 1993), but compounds with As—O—Si(R)$_2$—O—As bridging units have not been characterized previously. Additionally, all the reported As—O—$SiMe_3$ compounds contained arsenic in the +V oxidation state with tetra-, penta- or hexacoordination. We report here the structure of 3,3,7,7,10,10-hexaphenyl-2,4,6,8,9,11-hexaoxa-1,5-diarsa-3,7,10-trisilabicyclo[3.3.3]undecane, (I), in which the As atoms are tricoordinate and in the +III oxidation state.



(I)

Compound (I) contains two trigonal pyramidally coordinated AsO_3 units and three tetrahedrally coordinated SiO_2C_2 moieties arranged in a cage structure (Fig. 1). When viewed along the axis passing through both As atoms (Fig. 2), the AsO_3 units adopt a conformation with O*n*1—As1···As2—O*n*2 (*n* = 1–3) in the narrow range 36.4 (3)–37.7 (3)°; a perfectly staggered conformation with O—As···As—O torsion angles close to 60° is precluded by the constraints imposed by the $SiPh_2$ groups. The O—As—O angles vary between 97.72 (17) and 100.64 (18)° [mean 98.9 (10)°]. The O—Si—O angles exhibit less variation, with values of 111.7 (2), 110.9 (2) and 110.8 (2)° [mean 111.1 (4)°]. The six As—O—Si angles vary between 136.4 (2) and 140.4 (2)° [mean 139.1 (3)°]. The As—O—Si angles are somewhat larger in (I) than in the trimethylsiloxy derivatives of aminoarsenates $(Me_3SiO)_3AsO.(Me_3SiO)_3As(NMe)$ [135.3 (1), 132.3 (1) and 132.2 (1)°] and $(Me_3SiO)_2(PhNH)AsO$ [131.3 (2) and 131.6 (2)°, and 129.6 (2) and 132.0 (2)°, for the two crystallographically independent molecules in the asymmetric unit] (Baier, Paul & Schmidbaur, 1993) or the triarsazene compound $[(Me_3SiO)_2AsN]_3$, which has angles between 128.5 (4) and 136.0 (3)° (Baier, Bissinger & Schmidbaur, 1993).

The range of As—O(Si) bond lengths in the previously reported As—O—$SiMe_3$-containing compounds, in which arsenic may be four-, five- or six-coordinate, is notably large, *e.g.* 1.680 (3) Å for four-coordinate As in $(Me_3SiO)_8As_4O_6$ (Baier, Bissinger & Schmidbaur, 1992) to 1.780 (2) Å for five-coordinate As in $(Me_3SiO)_3AsO.(Me_3SiO)_3As(NMe)$ (Baier, Paul & Schmidbaur, 1993). In (I), the As—O(Si) distances are between 1.740 (4) and 1.781 (3) Å [mean 1.760 (15) Å].

The Si—O bond lengths in (I) fall within a narrow range, from 1.628 (4) to 1.643 (4) Å [mean 1.637 (5) Å]. The compound $Ph_2Si(OH)_2$ has three independent molecules in the asymmetric unit and there are two reports of the structure; one of these (Fawcett, Camerman & Camerman, 1977) has the Si—O distances in the narrow range 1.630–1.636 Å [mean 1.633 (2) Å] and the other (Párkányi & Bocelli, 1978) reports the Si—O distances to be in the range 1.636–1.649 Å [mean 1.642 (4) Å]. In $(Me_3SiO)_3AsO.(Me_3SiO)_3As(NMe)$, the Si—O distances are all between 1.646 (2) and 1.669 (2) Å (Baier, Paul & Schmidbaur, 1993) but in $[(Me_3SiO)_2AsN]_3$ they are in the large range 1.646 (5)–1.732 (6) Å (Baier, Bissinger & Schmidbaur, 1993). There are no unusual structural features associated with the $SiPh_2$ units in (I), with mean Si—C_{arom} 1.859 (7) and mean aromatic C—C 1.377 (16) Å. A view of the molecular packing has been deposited; intermolecular contacts are of the van der Waals type.

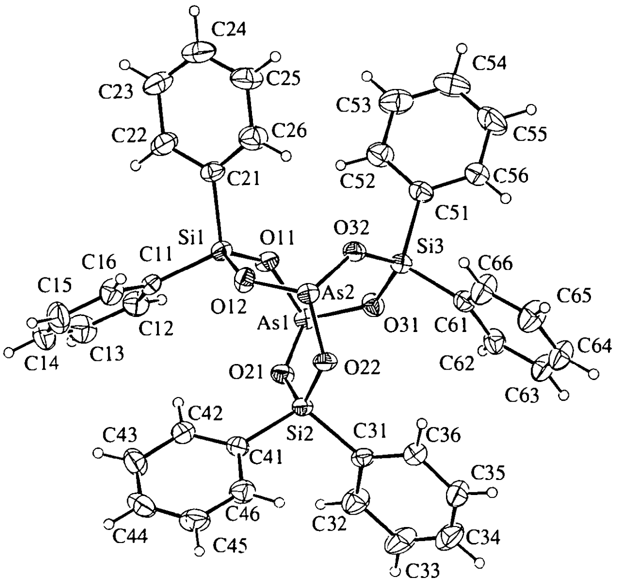


Fig. 1. A view of (I) with the numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

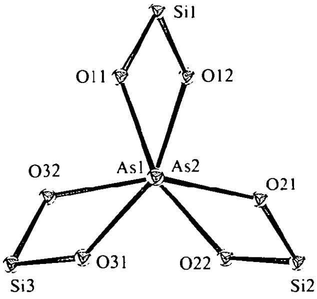


Fig. 2. A drawing of the $As_2Si_3O_6$ cage in (I) viewed along the As1···As2 vector. Atom As2 is directly behind As1. Displacement ellipsoids are drawn at the 30% probability level.

Experimental

Crystals of (I) were synthesized by the method of Chamberland & MacDairmid (1960). Crystals suitable for X-ray analysis were obtained by recrystallization from an ether/dichloromethane/hexane solution (1:1:1).

Crystal data

$C_{36}H_{30}As_2O_6Si_3$
$M_r = 792.72$

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å

Monoclinic
$P2_1$
$a = 8.2444\,(15)$ Å
$b = 10.7401\,(13)$ Å
$c = 20.049\,(3)$ Å
$\beta = 95.057\,(14)°$
$V = 1768.3\,(5)$ Å^3
$Z = 2$
$D_x = 1.489$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 9.55–15.20°$
$\mu = 2.03$ mm^{-1}
$T = 294\,(1)$ K
Plate
0.38 × 0.34 × 0.06 mm
Colorless

Data collection

Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: four ψ-scans at 4° steps (North, Phillips & Mathews, 1968)
$T_{\min} = 0.195$, $T_{\max} = 0.535$
4323 measured reflections
4066 independent reflections

3410 observed reflections $[I > 3\sigma(I)]$
$R_{\text{int}} = 0.008$
$\theta_{\max} = 26.91°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 25$
3 standard reflections
frequency: 120 min
intensity decay: 1.4%

Refinement

Refinement on F
$R = 0.029$
$wR = 0.038$
$S = 1.06$
3410 reflections
424 parameters
H atoms riding, C—H 0.95 Å
$w = 1/[\sigma^2(F) + 0.0008F^2]$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.54$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}

Extinction correction: Larson (1970)
Extinction coefficient: 1441 (379)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)
Absolute configuration: comparative refinement (see below); Rogers (1981) parameter = 1.0

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
As1	0.33020 (6)	0.0000	0.31718 (2)	0.0400 (2)
As2	0.64413 (6)	0.18124 (6)	0.19415 (2)	0.0392 (2)
Si1	0.71754 (15)	0.00730 (15)	0.32087 (6)	0.0407 (6)
Si2	0.36687 (16)	−0.02080 (13)	0.16132 (6)	0.0388 (6)
Si3	0.36969 (17)	0.28829 (14)	0.28018 (7)	0.0413 (6)
O11	0.5354 (4)	0.0264 (4)	0.34659 (16)	0.0464 (19)
O12	0.7243 (5)	0.0613 (4)	0.24528 (18)	0.0508 (19)
O21	0.3601 (4)	−0.0648 (3)	0.23908 (16)	0.0452 (18)
O22	0.4698 (4)	0.1090 (3)	0.15587 (18)	0.0454 (17)
O31	0.2818 (4)	0.1542 (3)	0.29057 (19)	0.048 (2)
O32	0.5536 (4)	0.2690 (3)	0.25649 (17)	0.0453 (17)
C11	0.7698 (6)	−0.1605 (5)	0.3179 (3)	0.046 (3)
C12	0.6846 (8)	−0.2524 (6)	0.3483 (3)	0.058 (3)
C13	0.7299 (11)	−0.3773 (7)	0.3440 (4)	0.075 (5)
C14	0.8574 (10)	−0.4103 (7)	0.3098 (4)	0.078 (5)
C15	0.9431 (9)	−0.3222 (8)	0.2789 (4)	0.079 (4)
C16	0.9006 (7)	−0.1974 (6)	0.2832 (3)	0.061 (3)
C21	0.8671 (6)	0.0914 (5)	0.3798 (3)	0.046 (3)
C22	0.9648 (8)	0.0268 (7)	0.4265 (3)	0.065 (4)
C23	1.0765 (10)	0.0895 (9)	0.4714 (4)	0.084 (5)
C24	1.0908 (9)	0.2154 (8)	0.4688 (4)	0.081 (5)
C25	0.9970 (10)	0.2806 (8)	0.4221 (4)	0.080 (4)
C26	0.8879 (9)	0.2193 (7)	0.3779 (4)	0.069 (4)
C31	0.1610 (6)	0.0104 (6)	0.1186 (2)	0.044 (2)
C32	0.0468 (8)	−0.0836 (7)	0.1152 (4)	0.067 (4)
C33	−0.1080 (8)	−0.0638 (9)	0.0831 (4)	0.084 (5)
C34	−0.1480 (8)	0.0462 (9)	0.0556 (4)	0.079 (5)
C35	−0.0360 (9)	0.1419 (7)	0.0565 (4)	0.070 (4)
C36	0.1160 (7)	0.1235 (6)	0.0899 (3)	0.055 (3)
C41	0.4648 (6)	−0.1526 (5)	0.1198 (3)	0.045 (2)
C42	0.5556 (7)	−0.2416 (6)	0.1556 (3)	0.055 (3)
C43	0.6291 (8)	−0.3381 (6)	0.1234 (4)	0.074 (4)
C44	0.6158 (8)	−0.3430 (6)	0.0544 (4)	0.072 (4)
C45	0.5244 (9)	−0.2583 (8)	0.0181 (3)	0.071 (4)
C46	0.4499 (8)	−0.1640 (6)	0.0498 (3)	0.060 (3)
C51	0.3939 (6)	0.3780 (5)	0.3599 (3)	0.048 (3)
C52	0.4879 (8)	0.3348 (6)	0.4158 (3)	0.060 (3)
C53	0.5023 (10)	0.4012 (8)	0.4740 (3)	0.079 (5)
C54	0.4225 (11)	0.5118 (10)	0.4790 (3)	0.088 (5)
C55	0.3287 (11)	0.5566 (7)	0.4255 (4)	0.086 (5)
C56	0.3143 (8)	0.4907 (7)	0.3660 (3)	0.067 (4)
C61	0.2415 (6)	0.3742 (5)	0.2155 (2)	0.045 (3)
C62	0.0745 (7)	0.3520 (5)	0.2025 (3)	0.054 (3)
C63	−0.0170 (7)	0.4197 (6)	0.1541 (3)	0.062 (3)
C64	0.0525 (8)	0.5079 (8)	0.1182 (3)	0.070 (4)
C65	0.2161 (8)	0.5326 (7)	0.1294 (4)	0.077 (4)
C66	0.3091 (7)	0.4655 (7)	0.1774 (4)	0.070 (4)

Table 2. *Selected geometric parameters* (Å, °)

As1—O11	1.764 (3)	Si1—C21	1.864 (5)
As1—O21	1.751 (3)	Si2—O21	1.635 (4)
As1—O31	1.775 (3)	Si2—O22	1.641 (4)
As2—O12	1.740 (4)	Si2—C31	1.864 (5)
As2—O22	1.750 (3)	Si2—C41	1.862 (5)
As2—O32	1.781 (3)	Si3—O31	1.633 (4)
Si1—O11	1.643 (4)	Si3—O32	1.641 (4)
Si1—O12	1.628 (4)	Si3—C51	1.862 (5)
Si1—C11	1.855 (6)	Si3—C61	1.846 (5)
O11—As1—O21	98.99 (16)	O22—Si2—C31	105.8 (2)
O11—As1—O31	97.72 (17)	O22—Si2—C41	111.7 (2)
O21—As1—O31	98.51 (17)	C31—Si2—C41	110.1 (2)
O12—As2—O22	100.64 (18)	O31—Si3—O32	110.9 (2)
O12—As2—O32	98.00 (17)	O31—Si3—C51	111.2 (2)
O22—As2—O32	99.42 (16)	O31—Si3—C61	107.3 (2)
O11—Si1—O12	110.8 (2)	O32—Si3—C51	106.4 (2)
O11—Si1—C11	110.6 (2)	O32—Si3—C61	110.2 (2)
O11—Si1—C21	108.2 (2)	C51—Si3—C61	110.9 (2)
O12—Si1—C11	106.8 (2)	As1—O11—Si1	138.7 (2)
O12—Si1—C21	110.0 (2)	As2—O12—Si1	140.4 (2)
C11—Si1—C21	110.4 (2)	As1—O21—Si2	139.2 (2)
O21—Si2—O22	111.7 (2)	As2—O22—Si2	139.2 (2)
O21—Si2—C31	112.6 (2)	As1—O31—Si3	140.4 (2)
O21—Si2—C41	105.0 (2)	As2—O32—Si3	136.4 (2)
O11—As1—As2—O12	36.7 (3)	O31—As1—As2—O32	37.7 (3)
O21—As1—As2—O22	36.4 (3)		

The compound crystallized in the monoclinic system; space groups $P2_1$ or $P2_1/m$ were indicated by the systematic absences. $P2_1$ was chosen from analysis of Patterson vectors and confirmed by the successful refinement. The structure was solved by Patterson heavy-atom methods and *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Fig. 1 was prepared using *ORTEP*II (Johnson, 1976) as implemented in *PLATON* (Spek, 1994*a*). Examination of the structure with *PLATON* showed that there were no solvent-accessible voids in the crystal lattice. Refinement with the opposite direction of the polar axis converged with higher R and wR values for the observed data (0.033 and 0.041, respectively).

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET*4 and *CELDIM* in *CAD-4-PC Software* (Enraf–Nonius, 1992). Data reduction: *DATRD*2 in *NRCVAX*94. Program(s) used to refine structure: *NRCVAX*94. Molecular graphics: *NRCVAX*94, *PLATON* and *PLUTON* (Spek 1994*b*). Software used to prepare material for publication: *NRCVAX*94.

GF thanks NSERC (Canada) for Grants in Aid of Research.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, and a molecular packing diagram, have been deposited with the IUCr (Reference: BK1128). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). C**51**, 2315–2317

(−)-(1*S*,3*R*,4*R*,6*S*)-4,7,7-Trimethyl-5-oxobicyclo[4.1.0]heptane-3-carbonitrile

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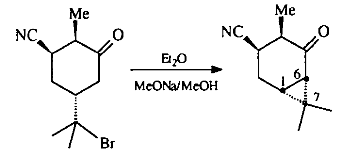
Abstract

The crystal structure of the title compound, $C_{11}H_{15}NO$, consists of discrete molecules held together by van der Waals interactions. The molecule contains a three-membered cyclopropane ring fused with a six-membered cyclohexanone ring, with a fold angle along the common bond of 65.4 (1)°. The relative stereochemistry of the molecule is established.

Comment

Cocker, Grayson & Shannon (1995) have recently described a series of fused isoxazolo–lactam compounds obtained from β-cyano-α-methylcycloalkanones by regiospecific nitrosonation at the α-position by pentanenitrile in methanolic sodium hydroxide. The reaction pathway probably involves sequential cycloalkanone cleavage, isoxazole formation and lactamization. In the course of the study, (−)-(1*R*,2*R*,5*R*)-5-(1-bromo-1-methylethyl)-2-methyl-3-oxocyclohexanecarbonitrile, (1), was prepared, but its relative configuration could not be assigned on the basis of spectral results. We therefore undertook an X-ray structure determination of the derived cyanocaranone, (2), in order to determine the relative stereochemistry of these compounds unambiguously.



(1) (2)

The structure of (2) is shown in Fig. 1, which also indicates the atom-numbering scheme used. Although the absolute configuration could not be determined unambiguously from the refinement results, due to the absence of significant anomalous scattering, the relative stereochemistries at positions 1, 3, 4 and 6 are clearly revealed as *S*, *R*, *R* and *S*, respectively. The cyclopropane ring makes an angle of 65.4 (1)° with the mean plane formed by atoms C1, C2, C5 and C6 [maximum deviation 0.015 (1) Å]. The six-membered ring as a whole is non-planar and has a twisted chair conformation with torsion angles C6—C1—C2—C3 −23.7 (2), C1—C2—C3—C4 53.5 (2), C2—C3—C4—C5 −61.1 (2), C3—C4—C5—C6 40.7 (2), C4—C5—

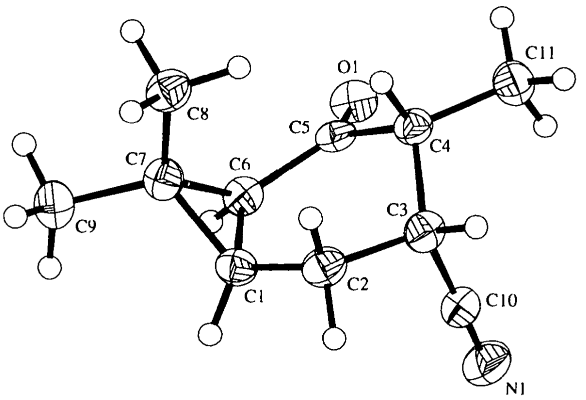


Fig. 1. The molecular structure of (2) showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by small circles.

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