

C6—C7—O7	110.9 (11)	O7—Si—C21	107.2 (5)
C8—C7—O7	108.6 (10)	O7—Si—C23	109.1 (6)
C3—C8—C7	104.7 (9)	O7—Si—C25	108.2 (6)
C3—C8—C9	110.9 (9)	C21—Si—C23	112.1 (7)
C3—C8—C19	116.4 (10)	C21—Si—C25	111.3 (7)
C7—C8—C9	107.6 (10)	C23—Si—C25	109.0 (8)
C7—C8—C19	109.4 (10)	Si—C21—C22	118.3 (11)
C9—C8—C19	107.6 (10)	Si—C23—C24	117.1 (16)
C8—C9—C10	123.4 (9)	Si—C25—C26	117.2 (15)
C8—C9—O9	118.5 (10)		
C15—C1—C2—C3	-59.3 (11)	C3—C2—O2—C20	21.0 (10)
C1—C2—C3—C8	109.7 (12)	C2—O2—C20—C4	-36.5 (11)
C2—C3—C8—C9	-77.7 (11)	O2—C20—C4—C3	35.8 (11)
C3—C8—C9—C10	-27.4 (9)	C20—C4—C3—C2	-22.7 (10)
C8—C9—C10—C11	53.4 (10)	C4—C3—C2—O2	2.0 (10)
C9—C10—C11—C15	49.8 (10)	C8—C3—C4—C5	-29.7 (10)
C10—C11—C15—C1	-115.7 (11)	C3—C4—C5—C6	25.1 (11)
C11—C15—C1—C2	65.1 (10)	C4—C5—C6—C7	-43.4 (12)
C14—C1—C15—C11	-62.8 (11)	C5—C6—C7—C8	65.7 (12)
C1—C15—C11—C12	49.7 (10)	C6—C7—C8—C3	-63.8 (11)
C15—C11—C12—C13	-6.3 (11)	C7—C8—C3—C4	46.9 (10)
C11—C12—C13—C14	-20.9 (12)	C20—C4—C5—O5	26.2 (11)
C12—C13—C14—C1	1.7 (11)	O4—C4—C5—O5	-96.9 (12)
C13—C14—C1—C15	39.9 (11)		

Because of the large thermal motion of the triethylsilyl group, the three ethyl chains have been refined with constraints on bond distances and angles, and have been kept isotropic, conferring to the high *R* factor and consequently a lack of precision in the results. However, all the H atoms of the hydroxyl groups were located in successive difference Fourier maps, allowing the building of the hydrogen-bond network. Their positional parameters were fixed (C—H = 1.00 Å). Isotropic temperature factors of the H atoms were assigned as $1.10 \times U_{eq}$ of the bonded atom.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Data reduction: *NONIUS* (Riche, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *R3M* (Riche, 1983), *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *ACTACIF* (Riche, 1992).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: PA1126). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The *endo-endo-syn* Diels–Alder Diadduct of Tetrachlorodimethoxycyclopentadiene with 1,6-Dioxacyclodeca-3,8-diene

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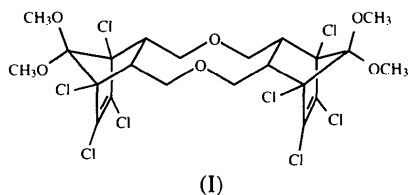
Abstract

In the title compound, *endo-endo-syn*-1,7,8,9,10,16,17,18-octachloro-19,19,20,20-tetramethoxy-4,13-dioxapentacyclo[14.2.1.1^{7,10}.0^{2,15}.0^{6,11}]icosa-8,17-diene, C₂₂H₂₄Cl₈O₆, the ten-membered ring adopts a chair–chair conformation. The near-zero endocyclic torsion angles of this ring at the ring fusion bonds have values of 0.0 (6) and -0.8 (4)°. The norbornene C=C bonds have lengths 1.320 (4) and 1.311 (5) Å. The four dioxolane O—C bonds have an average length of 1.413 (2) Å.

Comment

The crystal structures of a number of Diels–Alder-substituted cycloocta(e)nes and cyclohexa(e)nes have

been determined in our laboratory over the past three years (Garcia & McLaughlin, 1991; Garcia, Fronczek & McLaughlin, 1991*a,b,c*, 1992*a,b*; Garcia, McLaughlin & Fronczek, 1991*a,b*; Li, Fronczek & McLaughlin, 1992; Garcia, Morales, Fronczek & McLaughlin, 1994; Garcia & Fronczek, 1994) as part of a study of conformation-dependent π - σ - π electronic interactions (Garcia, Fronczek & McLaughlin, 1991*a*; Garcia & McLaughlin, 1991; Garcia & Fronczek, 1994). The crystal structure determination of the title compound, (I), is part of an ongoing program of structure analyses of some new derivatives bearing the dioxacyclodecane moiety.



Structural data for *endo-endo-anti*-1,6,7,8,9,14,15,16-octachloro-17,17,18,18-tetramethoxypentacyclo[12.2.1-1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene (Garcia, McLaughlin & Fronczek, 1991*a*), *endo-endo-anti*-17,17,18,18-tetramethoxypentacyclo[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene (Garcia, McLaughlin & Fronczek, 1991*a*), *endo-endo-anti*-1,6,7,8,9,14,15,16-octachloro-17,17-dimethoxypentacyclo[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene (Garcia, Fronczek & McLaughlin, 1992*b*), *endo*-1,10,11,12-tetrachloro-13,13-dimethoxytricyclo[8.2.1.0^{2,9}]trideca-5,11-diene (Li, Fronczek & McLaughlin, 1992), *endo-endo-syn*-16,16-dimethoxy-1,5,6,7,8,12,13,14,15,15-decachloropentacyclo[10.2.1.1^{5,8}.0^{2,11}.0^{4,9}]hexadeca-6,13-diene (Garcia & Fronczek, 1994), *endo-endo-syn*-1,5,6,7,8,12,13,14-octachloro-15,15,16,16-tetramethoxypentacyclo[10.2.1.1^{5,8}.0^{2,11}.0^{4,9}]hexadeca-6,13-diene (Garcia & Fronczek, 1994) and *trans*-1,6-cyclodecanediol (Ermer, Dunitz & Bernal, 1973) are in agreement with those of the title compound.

The dioxacyclodecane ring has a chair-chair conformation (Hendrickson, 1967*b*) with bond angles θ_1 [C2—C1—C8, C6—C5—C4], θ_2 [C1—C8—O2, C5—C4—O1], θ_3 [C8—O2—C7, C4—O1—C3], θ_4 [O2—C7—C6, O1—C3—C2], and θ_5 [C7—C6—C5, C3—C2—C1] with the following values: $\theta_1 = 118.3$ (2), 117.7 (3); $\theta_2 = 109.9$ (2), 106.7 (3); $\theta_3 = 115.0$ (2), 114.0 (2); $\theta_4 = 109.4$ (2), 108.7 (3); $\theta_5 = 117.8$ (3), 118.4 (2) $^\circ$. These are distorted from the theoretical value (Hendrickson, 1967*b*) of the long-chair form, $\theta_1 = \theta_2 = \theta_3 = \theta_4 = \theta_5 = 118^\circ$, as well as from the experimental values $\theta_1 = \theta_2 = \theta_5 = 118^\circ$ and $\theta_3 = \theta_4 = 115^\circ$ (Hendrickson, 1967*a*). The torsion angles ω_1 [C3—C2—C1—C8, C7—C6—C5—C4], ω_2 [C2—C1—C8—O2, C6—C5—C4—O1], ω_3 [C1—C8—O2—C7, C5—C4—O1—C3], ω_4 [C8—O2—C7—C6, C4—O1—C3—C2], and ω_5 [O2—C7—

C6—C5, O1—C3—C2—C1] have the following values: $\omega_1 = 0.0$ (6), -0.8 (4); $\omega_2 = -71.2$ (3), -68.5 (3); $\omega_3 = 145.4$ (4), 153.3 (3); $\omega_4 = -158.3$ (3), -163.0 (3); $\omega_5 = 82.0$ (3), 75.8 (3) $^\circ$. These are distorted from the torsion angles of the symmetrical long-chair conformation (of cyclodecane), $\omega_1 = \pm 0$, $\omega_2 = \pm 112.2$, $\omega_3 = 72.7$, $\omega_4 = \pm 72.7$ and $\omega_5 = \pm 112.2^\circ$ (Hendrickson, 1967*b*).

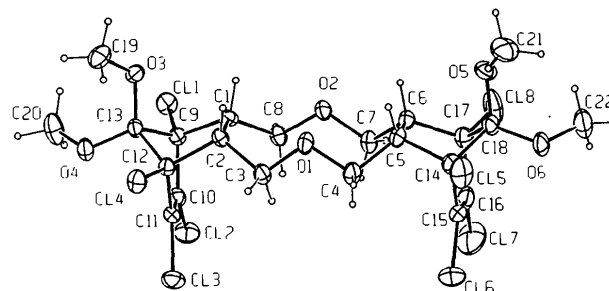


Fig. 1. ORTEP drawing (Johnson, 1965) of title molecule representing heavy atoms as 30% probability ellipsoids and H atoms as circles of arbitrary radii.

Experimental

Crystals suitable for analysis were prepared by reacting 1,6-dioxacyclodeca-3,8-diene (Reppe, 1955) with 1,2,3,4-tetrachloro-5,5-dimethoxycyclopenta-1,3-diene (Newcomer & McBee, 1949; McBee, Crain, Belohlav & Braendlin, 1962), following a modification of the method of Akhtar, Fray & Yarrow (1968). Crystals (m.p. 557–559 K) were grown by slow cooling of a *p*-xylene solution.

Crystal data

C₂₂H₂₄Cl₈O₆
 $M_r = 668.1$
 Orthorhombic
Pbca
 $a = 14.3565$ (7) Å
 $b = 14.8021$ (8) Å
 $c = 25.612$ (2) Å
 $V = 5442.8$ (9) Å³
 $Z = 8$
 $D_x = 1.630$ Mg m⁻³

Mo K α radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 25 reflections
 $\theta = 10$ –12 $^\circ$
 $\mu = 0.869$ mm⁻¹
 $T = 295$ K
 Needle
 0.42 \times 0.17 \times 0.15 mm
 Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω -2 θ scans
 Absorption correction: empirical
 $T_{\min} = 0.98$, $T_{\max} = 1.00$
 5316 measured reflections
 4769 independent reflections

3036 observed reflections [$I > 3\sigma(I)$]
 $\theta_{\max} = 25^\circ$
 $h = 0 \rightarrow 17$
 $k = 0 \rightarrow 17$
 $l = 0 \rightarrow 30$
 3 standard reflections
 frequency: 120 min
 intensity decay: <1%

Refinement

Refinement on F
 $R = 0.035$
 $wR = 0.037$

$(\Delta/\sigma)_{\max} < 0.01$
 $\Delta\rho_{\max} = 0.27$ e Å⁻³
 $\Delta\rho_{\min} = -0.10$ e Å⁻³

S = 1.468

3036 reflections

373 parameters

 $w = 4F_o^2/[\sigma^2(I) + (0.02F_o)^2]$

Atomic scattering factors
from *International Tables*
for *X-ray Crystallography*
(1974, Vol. IV)

C11—C9—C1	113.4 (2)	C12—C10—C9	124.1 (2)
C11—C9—C10	115.0 (2)	C12—C10—C11	128.6 (3)
C11—C9—C13	117.5 (2)	C9—C10—C11	107.3 (3)
C1—C9—C10	108.3 (2)	C13—C11—C10	127.7 (2)
C1—C9—C13	101.9 (2)	C13—C11—C12	124.3 (2)
C10—C9—C13	99.0 (2)	C10—C11—C12	108.0 (3)
C2—C1—C8	118.3 (2)	C14—C12—C2	114.6 (2)
C2—C1—C9	102.7 (2)	C14—C12—C11	114.0 (2)
C8—C1—C9	112.3 (2)	C14—C12—C13	116.2 (2)
C1—C2—C3	118.4 (2)	C2—C12—C11	109.1 (2)
C1—C2—C12	101.8 (2)	C2—C12—C13	102.1 (2)
C3—C2—C12	111.1 (3)	C11—C12—C13	99.2 (2)
O1—C3—C2	108.7 (3)	O3—C13—O4	113.4 (2)
O1—C4—C5	106.7 (3)	O3—C13—C9	106.7 (2)
C4—C5—C6	117.7 (3)	O3—C13—C12	117.6 (2)
C4—C5—C14	112.4 (3)	O4—C13—C9	118.9 (2)
C6—C5—C14	102.1 (2)	C15—C16—C17	107.2 (3)
C5—C6—C7	117.8 (3)	C18—C17—C6	113.0 (2)
C5—C6—C17	102.1 (2)	C18—C17—C16	115.7 (2)
C7—C6—C17	112.7 (3)	C18—C17—C18	116.3 (2)
O2—C7—C6	109.4 (2)	C6—C17—C16	108.0 (3)
O2—C8—C1	109.9 (2)	C6—C17—C18	102.3 (3)
O4—C13—C12	107.9 (2)	C16—C17—C18	100.0 (3)
C9—C13—C12	90.8 (2)	O5—C18—O6	113.4 (3)
C15—C14—C5	114.5 (2)	O5—C18—C14	117.9 (3)
C15—C14—C15	114.5 (2)	O5—C18—C17	106.8 (3)
C15—C14—C18	116.3 (2)	O6—C18—C14	107.7 (3)
C5—C14—C15	108.7 (3)	O6—C18—C17	119.0 (3)
C5—C14—C18	101.6 (3)	C14—C18—C17	90.5 (2)
C4—O1—C3—C2	-163.0 (3)	C3—O1—C4—C5	153.3 (3)
C8—O2—C7—C6	-158.3 (3)	C7—O2—C8—C1	145.4 (4)
C8—C1—C2—C3	0.0 (6)	C2—C1—C8—O2	-71.2 (3)
C1—C2—C3—O1	75.8 (3)	O1—C4—C5—C6	-68.5 (3)
C4—C5—C6—C7	-0.8 (4)	C5—C6—C7—O2	82.0 (3)

H atoms were refined isotropically except for those of the methyl groups which were placed in calculated positions with C—H distances of 0.95 Å and $B_{\text{iso}} = 1.3B_{\text{eq}}$ of the bonded C atoms. Programs used include *MolEN* (Fair, 1990), *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and *ORTEP* (Johnson, 1965).

We thank DOE for support of this research through grant DE-AC03-76SF00098.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including torsion angles, have been deposited with the IUCr (Reference: BK1038). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

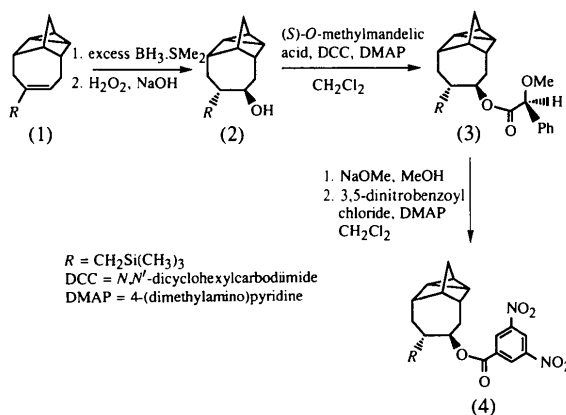
	x	y	z	B_{eq}
C11	0.29410 (6)	0.28750 (6)	0.54816 (3)	3.48 (2)
C12	0.42917 (6)	0.46375 (7)	0.55662 (3)	4.12 (2)
C13	0.34552 (6)	0.63282 (6)	0.47833 (4)	4.09 (2)
C14	0.14898 (6)	0.56787 (6)	0.42675 (3)	3.55 (2)
C15	0.50571 (8)	0.43897 (7)	0.17764 (4)	5.43 (2)
C16	0.64887 (9)	0.53442 (7)	0.25923 (6)	7.48 (3)
C17	0.74813 (8)	0.3747 (1)	0.33608 (5)	7.98 (4)
C18	0.66568 (8)	0.17414 (7)	0.30232 (4)	6.08 (2)
O1	0.3485 (2)	0.4149 (2)	0.32942 (8)	3.36 (5)
O2	0.4305 (2)	0.2812 (1)	0.39070 (8)	3.05 (5)
O3	0.1280 (1)	0.3496 (1)	0.47427 (8)	2.99 (5)
O4	0.1463 (1)	0.4685 (2)	0.53265 (8)	3.26 (5)
O5	0.5242 (2)	0.2141 (2)	0.21147 (9)	4.22 (6)
O6	0.6574 (2)	0.2931 (2)	0.18846 (9)	4.01 (5)
C1	0.3109 (2)	0.3441 (2)	0.4448 (1)	2.37 (6)
C2	0.2675 (2)	0.4209 (2)	0.4099 (1)	2.38 (6)
C3	0.3305 (2)	0.4709 (2)	0.3726 (1)	3.07 (7)
C4	0.4262 (2)	0.4421 (2)	0.2992 (1)	3.48 (7)
C5	0.4638 (2)	0.3580 (2)	0.2731 (1)	2.81 (7)
C6	0.5107 (2)	0.2847 (2)	0.3088 (1)	2.83 (7)
C7	0.5163 (2)	0.3036 (2)	0.3670 (1)	3.03 (7)
C8	0.4134 (2)	0.3240 (2)	0.4392 (1)	2.79 (7)
C9	0.2847 (2)	0.3748 (2)	0.5012 (1)	2.40 (6)
C10	0.3390 (2)	0.4595 (2)	0.5137 (1)	2.59 (6)
C11	0.3058 (2)	0.5245 (2)	0.4838 (1)	2.55 (6)
C12	0.2268 (2)	0.4856 (2)	0.4516 (1)	2.47 (6)
C13	0.1858 (2)	0.4182 (2)	0.4926 (1)	2.68 (7)
C14	0.5426 (2)	0.3794 (2)	0.2335 (1)	3.13 (7)
C15	0.6225 (2)	0.4226 (2)	0.2624 (1)	3.80 (8)
C16	0.6610 (2)	0.3607 (3)	0.2919 (1)	4.00 (8)
C17	0.6078 (2)	0.2742 (2)	0.2832 (1)	3.20 (7)
C18	0.5848 (2)	0.2837 (2)	0.2238 (1)	3.29 (7)
C19	0.0334 (3)	0.3733 (3)	0.4630 (2)	4.21 (9)
C20	0.1233 (3)	0.4237 (3)	0.5789 (1)	5.3 (1)
C21	0.4884 (3)	0.2117 (3)	0.1595 (2)	6.3 (1)
C22	0.7194 (3)	0.2198 (3)	0.1812 (2)	5.8 (1)

Table 2. Selected geometric parameters (Å, °)

C11—C9	1.770 (3)	C1—C2	1.575 (4)
C12—C10	1.700 (3)	C1—C8	1.507 (4)
C13—C11	1.707 (3)	C1—C9	1.561 (4)
C14—C12	1.771 (3)	C2—C3	1.509 (4)
C15—C14	1.761 (3)	C2—C12	1.548 (4)
C16—C15	1.699 (4)	C4—C5	1.512 (5)
C17—C16	1.700 (4)	C5—C6	1.571 (4)
C18—C17	1.767 (3)	C5—C14	1.553 (4)
O1—C3	1.406 (4)	C6—C7	1.518 (4)
O1—C4	1.416 (4)	C6—C17	1.548 (5)
O2—C7	1.414 (4)	C9—C10	1.510 (4)
O2—C8	1.417 (4)	C9—C13	1.575 (4)
O3—C13	1.391 (4)	C10—C11	1.320 (4)
O3—C19	1.432 (4)	C11—C12	1.515 (4)
O4—C13	1.389 (4)	C12—C13	1.564 (4)
O4—C20	1.396 (4)	C14—C15	1.507 (5)
O5—C18	1.384 (4)	C14—C18	1.562 (5)
O5—C21	1.428 (5)	C15—C16	1.311 (5)
O6—C18	1.388 (4)	C16—C17	1.507 (5)
O6—C22	1.415 (5)	C17—C18	1.564 (5)
C3—O1—C4	114.0 (2)	C15—C14—C18	99.6 (3)
C7—O2—C8	115.0 (2)	C16—C15—C14	124.0 (3)
C13—O3—C19	117.1 (2)	C16—C15—C16	127.9 (3)
C13—O4—C20	118.0 (3)	C14—C15—C16	107.9 (3)
C18—O5—C21	117.3 (3)	C17—C16—C15	127.6 (3)
C18—O6—C22	118.7 (3)	C17—C16—C17	125.0 (3)

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preparation and NMR analysis of Mosher's esters (Dale, Dull & Mosher, 1969; Dale & Mosher, 1973) and mandelic esters (Dale & Mosher, 1968; Bonner, 1951), for racemic and enantiomerically enriched materials. We have already established the absolute stereochemistry of 5-phenyldeltacylene by the X-ray structural analysis of its Mosher's ester derivative (Lautens, Lautens & Lough, 1991). In this case we were unable to grow suitable single crystals of the Mosher's ester derivative (3). We therefore carried out the determination of the structure of the corresponding 3,5-dinitrobenzoate (4) and used the X-ray data to confirm the absolute stereochemistry.



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4-[(Trimethylsilyl)methyl]tetracyclo-[5.4.0.0^{6,10}.0^{9,11}]undec-3-yl 3,5-Dinitrobenzoate

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Abstract

The absolute stereochemistry of the title compound, $\text{C}_{22}\text{H}_{28}\text{N}_2\text{O}_6\text{Si}$, has been confirmed. The bond lengths and angles of the deltacyclane moiety are affected by strain.

Comment

The reaction between norbornadiene and a 2-substituted butadiene yields a substituted deltacyclene (1). Recently, we reported that a cobalt complex in the presence of catalytic quantities of a chiral bidentate phosphine promotes this higher order [4 + 2 + 2] cycloaddition with an enantiomeric excess greater than 70% (Lautens, Tam & Sood, 1993). The assignment of the absolute stereochemistry of the deltacyclene (1) was made on the basis of a well known empirical method, the

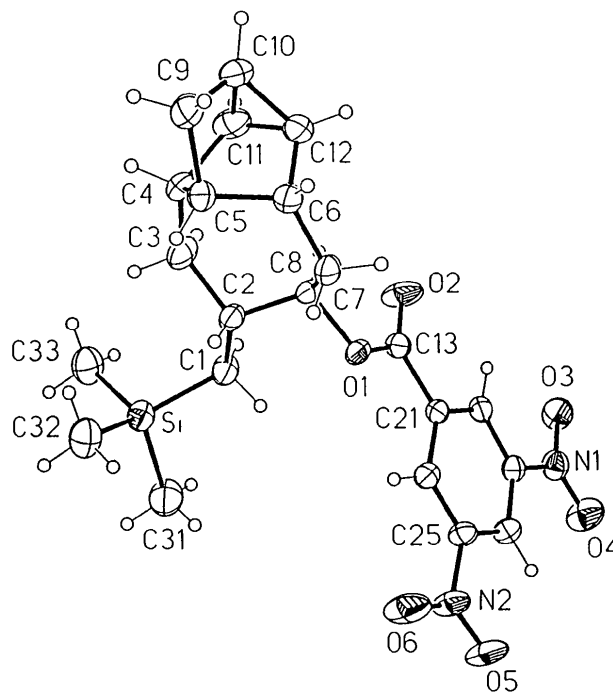


Fig. 1. View of the molecule with the crystallographic numbering scheme. Displacement ellipsoids are drawn at the 25% probability level and H atoms are shown as spheres of arbitrary radii for clarity.