*Refinement*

Refinement on $F^2$
$R(F) = 0.0612$
$wR(F^2) = 0.1734$
$S = 1.393$
1264 reflections
182 parameters
All H-atom parameters refined
Calculated weights
$w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.310$
$\Delta\rho_{\rm max} = 0.224$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.211$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| Cl1 | 0.35996 (10) | 0.01963 (4) | 0.2306 (2) | 0.0551 (5) |
| C1 | 0.6211 (4) | 0.3608 (2) | −0.0293 (6) | 0.0504 (12) |
| C2 | 0.5537 (5) | 0.3244 (2) | 0.0372 (7) | 0.0594 (13) |
| C3 | 0.5904 (7) | 0.2804 (2) | 0.0039 (9) | 0.078 (2) |
| C4 | 0.6995 (8) | 0.2731 (3) | −0.0942 (11) | 0.096 (3) |
| C5 | 0.7648 (7) | 0.3094 (3) | −0.1633 (9) | 0.080 (2) |
| C6 | 0.7265 (5) | 0.3531 (2) | −0.1319 (7) | 0.0640 (15) |
| C7 | 0.5635 (4) | 0.4045 (2) | 0.0274 (7) | 0.0510 (13) |
| C8 | 0.4782 (4) | 0.3911 (2) | 0.1984 (6) | 0.0481 (12) |
| C9 | 0.4452 (5) | 0.3410 (2) | 0.1457 (10) | 0.0653 (15) |
| C10 | 0.5426 (5) | 0.3922 (3) | 0.3990 (9) | 0.061 (2) |
| C11 | 0.3649 (4) | 0.4197 (2) | 0.2042 (8) | 0.0537 (13) |
| N12 | 0.3864 (4) | 0.4677 (2) | 0.2559 (9) | 0.0627 (13) |

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

| | | | |
|---|---|---|---|
| C1—C2 | 1.377 (7) | C5—C6 | 1.364 (8) |
| C1—C6 | 1.378 (7) | C7—C8 | 1.536 (6) |
| C1—C7 | 1.481 (7) | C8—C11 | 1.515 (6) |
| C2—C3 | 1.366 (8) | C8—C10 | 1.518 (7) |
| C2—C9 | 1.490 (7) | C8—C9 | 1.551 (8) |
| C3—C4 | 1.398 (11) | C11—N12 | 1.466 (7) |
| C4—C5 | 1.367 (11) | | |
| C2—C1—C6 | 120.0 (5) | C1—C7—C8 | 103.7 (4) |
| C2—C1—C7 | 110.5 (4) | C11—C8—C10 | 111.1 (4) |
| C6—C1—C7 | 129.5 (5) | C11—C8—C7 | 113.2 (4) |
| C3—C2—C1 | 120.7 (6) | C10—C8—C7 | 110.8 (4) |
| C3—C2—C9 | 129.0 (6) | C11—C8—C9 | 109.2 (4) |
| C1—C2—C9 | 110.3 (5) | C10—C8—C9 | 109.4 (5) |
| C2—C3—C4 | 118.7 (7) | C7—C8—C9 | 102.7 (4) |
| C5—C4—C3 | 120.2 (7) | C2—C9—C8 | 103.1 (4) |
| C6—C5—C4 | 120.5 (7) | N12—C11—C8 | 113.5 (4) |
| C5—C6—C1 | 119.7 (6) | | |

All e.s.d.'s were estimated using the full covariance matrix. Data collection: Stoe Stadi diffractometer control. Cell refinement: local program. Data reduction: *SHELX*76 (Sheldrick, 1976). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*92 (Sheldrick, 1994). Molecular graphics: *PLUTON*, *PLATON* (Spek, 1990, 1992). Software used to prepare material for publication: *SHELXL*92.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71755 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1078]

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*Acta Cryst.* (1994). C**50**, 729–732

# Dimethyl [3,3,4,4,5,5-Hexamethyl-1(*E*),2(*E*)-cyclopentandiylidene]diacetate, $C_{17}H_{26}O_4$

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## Abstract

To establish the conformation of the diene system of the title compound, its structure was determined by X-ray analysis. The non-planar butadiene fragment is in a *gauche* conformation [torsion angle 44.8 (9)°]. The methoxycarbonyl groups are fully eclipsed with respect to the single bond in the diene system. The five-membered ring adopts a distorted envelope conformation with the apical C atom at position 3. In the cyclopentane ring, elongations of C—C bonds [1.587 (2) and 1.575 (2) Å] and contractions of C—C—C bond angles [99.9 (2), 104.0 (2), 103.3 (2)°] involving atoms at positions 3, 4 and 5 are observed.

## Comment

Several unconventional products have been isolated from the combination of 3,3,4,4,5,5-hexamethyl-1,2-bis(methylene)cyclopentane with cycloaddition partners, which normally react in a 1,2 manner with other conjugated dienes (Baran & Mayr, 1987, and references therein). Therefore, this diene has been employed as a probe for the study of cycloaddition mechanisms; the results of these investigations have been reviewed recently (Mayr, Baran & Heigl, 1991). Studies of muconic acid derivatives, structurally related to this diene and possessing known (*E*,*E*) configuration at the 1,3-diene termini, should enable the elucidation of the stereochemistry of these cycloadditions. To obtain unequivocal proof

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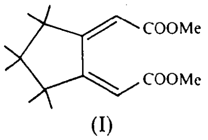
of its *E,E* configuration, the structure determination of one compound from the series, which was synthesized recently by Baran (1993), was undertaken. The structure and numbering scheme of the title compound (I) are presented in Fig. 1.



(I)

It is known that the conformation of a *cis*-butadiene system is governed by a delicate interplay between conjugation along the carbon chain, which favours the planar structure, and steric interaction between the terminal substituents, which favours a *gauche* structure. Theoretical studies (based on effects of dynamical electron correlation) on the structure of *cis*-butadiene have shown that the steric hindrance between terminal H atoms determines the twisting conformation of the diene system (torsion angle *ca* 40°) (Rice, Liu, Lee & Rohlfing, 1989). Nevertheless, in *cis*-butadiene, where positions 2 and 3 are fixed as in 3,3,4,4,5,5-hexamethyl-1,2-bis-(methylene)cyclopentane, the planarity of the diene system was confirmed (Mayr *et al.*, 1991). The title compound contains a non-planar butadiene system in a *gauche* conformation [C10=C11—C15=C16 44.8 (9)°]. The C15=C16—C17=O4 and C11=C10—C9=O2 fragments adopt the synperiplanar and the antiperiplanar conformation, respectively. The unusual antiperiplanar orientation of the C11=C10—C9=O2 chain (Leiserowitz, 1976) is possibly caused by dipole-dipole repulsion between the C9—O(1) and C(17)=O(4) groups, as well as by steric factors. The shortest C9···O4 [2.897 (2) Å] and C17···O1 [2.894 (2) Å] contacts, which are similar to those found in other compounds (Ramasubbu & Venkatesan, 1982; Ramasubbu, Rajaram & Venkatesan, 1982), confirm the existence of such intramolecular electrostatic interaction (Fig. 2). Thus, the twisted form of the diene system which is attributable to steric repulsions, is also affected by additional interactions between methoxycarbonyl groups. The geometric parameters of the C=CH—$COOCH_3$ fragments, *i.e.* their planarity, bond lengths and bond angles, are comparable to those found and calculated in similar compounds (Einspahr & Donohue, 1973; Rice *et al.*, 1989; Kveseth, Seip & Kohl, 1980; Caminati, Grassi & Bauder,1988). The orientations of the methoxycarbonyl groups with respect to the single bond in the diene system are defined by the torsion angles C9—C10=C11—C15 [0.9 (11)°] and C11—C15=C16—C17 [−1.1 (10)°].

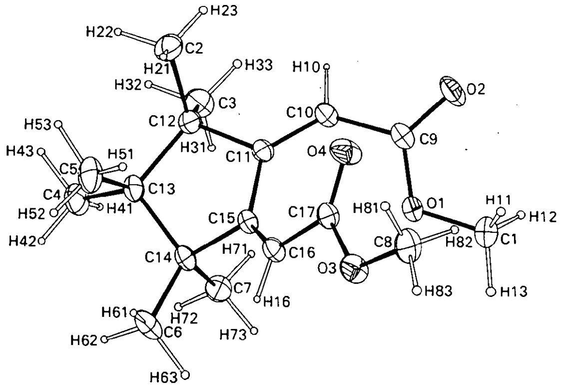


Fig. 1. The structure and numbering scheme of the title compound.

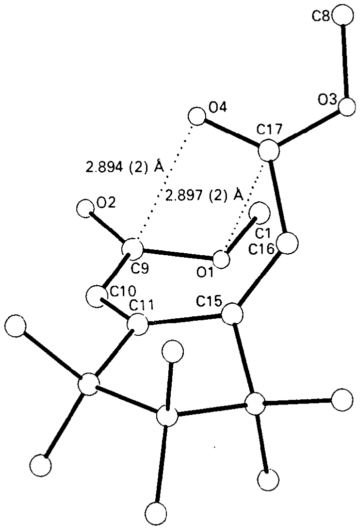


Fig. 2. A view of the molecule showing intramolecular interactions between the methoxycarbonyl groups.

The five-membered ring (in the sequence C15, C11, C12, C13 and C14) has a distorted envelope conformation [$Q_2$ = 0.430 (2) Å, $\varphi_2$ = 149.8 (3)°] with C14 on the flap [puckering parameters from Cremer & Pople (1975)]. Among the interesting features of the structure, there are the long C12—C13 [1.587 (2) Å] and C13—C14 [1.575 (2) Å] bond lengths and the contracted C15—C14—C13 [99.9 (2)°], C14—C13—C12 [104.0 (2)°] and C13—C12—C11 [103.3 (2)°] bond angles. These deformations may be explained by close intramolecular contacts between methyl groups (see Table 2) which induce steric repulsion. A similar deformation effect for a small ring with bulky substituents was found by us in 1,2,2,3,3,4,4,5-octamethyl-6,7,8-trioxabicyclo[3.2.1]octane (Jerzykiewicz, Dziewońska-Baran, Baran & Lis, 1993).

## Experimental

*Crystal data*

$C_{17}H_{26}O_4$
$M_r$ = 294.39

Cu $K\alpha$ radiation
$\lambda$ = 1.5418 Å

Monoclinic
$P2_1/c$
$a = 7.655$ (7) Å
$b = 11.436$ (8) Å
$c = 19.541$ (14) Å
$\beta = 107.45$ (7)°
$V = 1632$ (3) Å$^3$
$Z = 4$
$D_x = 1.198$ (3) Mg m$^{-3}$
$D_m = 1.18$ Mg m$^{-3}$
$D_m$ by flotation in KCl/H$_2$O

Cell parameters from 25 reflections
$\theta = 9$- 15°
$\mu = 0.642$ mm$^{-1}$
$T = 288$ (2) K
Platy shape
0.7 × 0.5 × 0.14 mm
Colourless
Crystal source: slow evaporation of ethanol solution

*Data collection*

Kuma KM-4 computer-controlled four-circle $\kappa$-axis diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.93$, $T_{\max} = 1.12$
3655 measured reflections
3042 independent reflections
2764 observed reflections [$F > 6\sigma(F)$]

$R_{\rm int} = 0.0149$ (289 reflections)
$\theta_{\max} = 75°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 14$
$l = -24 \rightarrow 23$
3 standard reflections monitored every 50 reflections intensity variation: −8%

*Refinement*

Refinement on $F$
$R = 0.035$
$wR = 0.031$
294 parameters
2459 reflections
All H-atom parameters refined
Calculated weights $w = 1/\sigma^2(F_o)$

$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.12$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.12$ e Å$^{-3}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

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

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

| | $x$ | $y$ | $z$ | $B_{\rm eq}$ |
|---|---|---|---|---|
| C1 | 0.78189 (25) | 0.31959 (18) | 0.60260 (9) | 5.05 (8) |
| C2 | 0.66786 (25) | 0.03906 (20) | 0.29429 (11) | 5.79 (10) |
| C3 | 0.53783 (23) | −0.08114 (15) | 0.37140 (11) | 4.72 (8) |
| C4 | 0.19877 (24) | −0.01166 (18) | 0.26832 (10) | 5.06 (8) |
| C5 | 0.34005 (27) | 0.16134 (19) | 0.23234 (9) | 5.56 (9) |
| C6 | 0.12320 (20) | 0.25247 (16) | 0.31863 (10) | 4.70 (8) |
| C7 | 0.22849 (20) | 0.09977 (16) | 0.41105 (9) | 3.91 (7) |
| C8 | 0.76525 (25) | 0.59905 (17) | 0.46460 (11) | 5.16 (8) |
| C9 | 0.83271 (18) | 0.18456 (14) | 0.51917 (8) | 3.35 (6) |
| C10 | 0.75464 (18) | 0.11666 (14) | 0.45365 (8) | 3.21 (6) |
| C11 | 0.60216 (17) | 0.13380 (13) | 0.39928 (7) | 2.82 (5) |
| C12 | 0.53518 (18) | 0.04262 (14) | 0.33955 (8) | 3.38 (6) |
| C13 | 0.33581 (19) | 0.08709 (14) | 0.29701 (8) | 3.47 (6) |
| C14 | 0.28096 (17) | 0.16820 (14) | 0.35239 (8) | 3.04 (5) |
| C15 | 0.46413 (16) | 0.22776 (13) | 0.38731 (7) | 2.78 (5) |
| C16 | 0.48252 (18) | 0.34296 (13) | 0.39515 (8) | 3.25 (6) |
| C17 | 0.65358 (19) | 0.41156 (13) | 0.42021 (8) | 3.21 (6) |
| O1 | 0.71143 (13) | 0.24988 (11) | 0.53890 (5) | 4.03 (5) |
| O2 | 0.99133 (14) | 0.17753 (11) | 0.55465 (7) | 4.99 (5) |
| O3 | 0.61427 (14) | 0.51887 (10) | 0.43836 (7) | 4.32 (5) |
| O4 | 0.80544 (14) | 0.38074 (11) | 0.42272 (7) | 4.90 (6) |

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

| | | | |
|---|---|---|---|
| O1—C1 | 1.440 (2) | C14—C15 | 1.524 (2) |
| O3—C8 | 1.444 (2) | C2—C12 | 1.534 (2) |
| O1—C9 | 1.335 (2) | C3—C12 | 1.544 (2) |
| O2—C9 | 1.207 (2) | C4—C13 | 1.529 (3) |
| O3—C17 | 1.337 (2) | C5—C13 | 1.531 (2) |
| O4—C17 | 1.202 (2) | C6—C14 | 1.531 (2) |
| C10—C9 | 1.463 (2) | C7—C14 | 1.537 (2) |
| C16—C17 | 1.478 (2) | C2· · ·C5 | 2.810 (3) |
| C11—C10 | 1.336 (2) | C3· · ·C4 | 2.879 (3) |
| C15—C16 | 1.329 (2) | C4· · ·C7 | 3.012 (3) |
| C11—C15 | 1.476 (2) | C5· · ·C6 | 2.892 (3) |
| C11—C12 | 1.534 (2) | C17· · ·O1 | 2.894 (2) |
| C12—C13 | 1.587 (2) | C9· · ·O4 | 2.897 (2) |
| C13—C14 | 1.575 (2) | | |
| C2—C12—C3 | 106.1 (2) | C13—C14—C15 | 99.9 (2) |
| C2—C12—C11 | 109.2 (2) | C14—C15—C11 | 105.3 (2) |
| C2—C12—C13 | 113.9 (2) | C14—C15—C16 | 123.1 (2) |
| C3—C12—C11 | 110.8 (2) | C11—C15—C16 | 131.1 (2) |
| C3—C12—C13 | 113.5 (2) | C12—C11—C15 | 108.3 (2) |
| C11—C12—C13 | 103.3 (2) | C12—C11—C10 | 121.2 (2) |
| C4—C13—C5 | 106.8 (2) | C15—C11—C10 | 130.2 (2) |
| C4—C13—C12 | 113.7 (2) | C11—C10—C9 | 130.4 (2) |
| C4—C13—C14 | 113.1 (2) | C10—C9—O2 | 123.0 (2) |
| C5—C13—C12 | 110.8 (2) | C10—C9—O1 | 114.4 (2) |
| C5—C13—C14 | 108.5 (2) | O2—C9—O1 | 122.6 (2) |
| C12—C13—C14 | 104.0 (2) | C9—O1—C1 | 116.5 (2) |
| C6—C14—C7 | 107.0 (2) | C15—C16—C17 | 128.1 (2) |
| C6—C14—C13 | 114.4 (2) | C16—C17—O4 | 127.4 (2) |
| C6—C14—C15 | 114.4 (2) | C16—C17—O3 | 109.1 (2) |
| C7—C14—C13 | 113.3 (2) | O4—C17—O3 | 123.4 (2) |
| C7—C14—C15 | 107.7 (2) | C17—O3—C8 | 117.2 (2) |
| C1—O1—C9—O2 | 4.6 (2) | C17—C16—C15—C11 | −1.1 (3) |
| C1—O1—C9—C10 | −179.2 (1) | C17—C16—C15—C14 | −172.3 (2) |
| O1—C9—C10—C11 | 23.3 (3) | C10—C11—C15—C16 | 44.8 (9) |
| C9—C10—C11—C15 | 0.9 (3) | C5—C13—C12—C2 | −23.0 (2) |
| C9—C10—C11—C12 | −172.9 (2) | C4—C13—C12—C3 | −24.3 (2) |
| C8—O3—C17—O4 | −2.3 (3) | C4—C13—C14—C7 | 48.3 (4) |
| C8—O3—C17—C16 | 179.8 (1) | C5—C13—C14—C6 | 43.5 (4) |
| O3—C17—C16—C15 | −163.9 (2) | | |

A parallelepiped crystal fragment was cut from a larger one and mounted in a Lindemann capillary to prevent its sublimation. Data collection was performed using Kuma KM-4 software. During the data collection, intensities of the standard reflections dropped by up to 92% of their initial values. Data were corrected for crystal decay. The structure was solved with *SHELXS*86 (Sheldrick, 1990) and refined using *XTL*/*XTLE* (Syntex, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71783 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1052]

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# Intermolecular Hydrogen-Bonded Structure of (1*R*)-(+)-Camphor Semicarbazone

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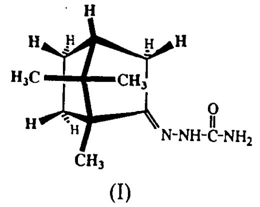
## Abstract

The first crystal structure of a rigid asymmetric semicarbazone, $C_{11}H_{19}N_3O$ {hydrazinecarboxamide, (1*R*)-(+)-1,7,7-trimethylbicyclo[2.2.1]hept-2-ylene-}, is described. [For a discussion of CAS/IUPAC nomenclature of semicarbazones see Grasselli & Weast (1989). *Handbook of Data on Organic Compounds*, 2nd ed., Vol. I, Introduction, §§190, 252. Boca Raton, Florida: CRC Press.] Crystalline (1*R*)-(+)-camphor semicarbazone (I) was found to have a fused hydrogen-bonded polymeric non-centrosymmetric structure. In each monomeric unit of (I), the hydrazidic H atom and one amidic H atom are *syn* to the carbonyl O atom; these atoms are involved in the intermolecular hydrogen bonding. The carbonyl O atom of each molecule is hydrogen bonded to the hydrazidic H atom of an adjacent molecule and the *syn*-amidic H atom of a different adjacent molecule to form a chain of fused eight-membered rings, the conformation of each ring being distorted from planarity. The hydrazidic hydrogen-bond geometry is N—H = 0.83 (4), H···O = 2.31 (4), N···O = 3.137 (4) Å and N—H···O = 177 (4)°. For the amidic hydrogen bond, N—H = 0.90 (4), H···O = 2.05 (4), N···O = 2.933 (4) Å and N—H···O = 167 (4)°.

## Comment

The abnormally high IR C═O frequencies exhibited by semicarbazones in the solid state, investigated by correlating a combination of Raman spectroscopic and X-ray crystallographic data, have been traced to vibrational coupling of C═O in the solid-state hydrogen-bonded network (Kolb, Dantzman, Kozenski & Strommen, 1993; see also discussion in Kolb, Robinson & Meyers, 1994). In that study, the X-ray crystal structures of 14 aliphatic and aromatic aldehyde and ketone semicarbazones typically showed a pattern of polymeric intermolecularly hydrogen-bonded fused eight-membered rings (Kolb, Stupar, Janota & Duax, 1989; Naik & Palenik, 1974; Etter, Britton & Videnova-Adrabinska, 1990; Pangborn, Galitsky, Duax & Kolb, 1991). In this context we chose next to examine (1*R*)-(+)-camphor semicarbazone (I), whose atypical rigid-bicyclic asymmetric molecular structure might give rise to a different hydrogen-bonding pattern. No X-ray structure of a rigid asymmetric semicarbazone had been reported prior to our initial report of these results (Kolb, Meyers & Robinson, 1993).



(I)

The preparation of (1*R*)-(+)-camphor semicarbazone (I) from the reaction of (1*R*)-(+)-camphor with semicarbazide (Kolb, Stupar, Janota & Duax, 1989) provided colorless crystals (from ethanol–water), m.p. 510–515 K (uncorrected). The structure of a monomeric unit is shown in Fig. 1.

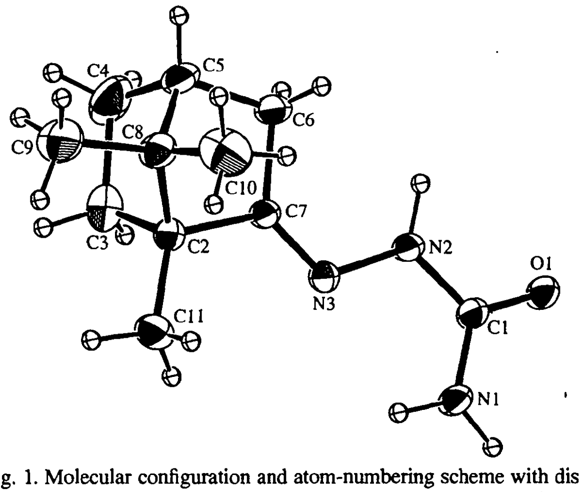


Fig. 1. Molecular configuration and atom-numbering scheme with displacement ellipsoids at the 30% probability level. H atoms are shown as unlabeled isotropic spheres of arbitrary radii.

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