

C(9B)	0.3605 (4)	0.2848 (2)	0.9754 (3)	0.067 (1)
C(10B)	0.2390 (4)	0.3240 (2)	0.8792 (3)	0.070 (1)
C(11B)	0.1847 (5)	0.2927 (2)	0.7477 (3)	0.073 (1)
C(12B)	0.1503 (4)	0.2169 (2)	0.7479 (3)	0.059 (1)

Table 4. Selected geometric parameters (Å, °) and hydrogen-bonding geometry (Å, °) for (3a)

O(1A)—C(6A)	1.435 (5)	O(1B)—C(6B)	1.434 (4)	
C(1A)—C(2A)	1.534 (5)	C(1B)—C(2B)	1.544 (6)	
C(1A)—C(3A)	1.455 (5)	C(1B)—C(3B)	1.461 (6)	
C(1A)—C(5A)	1.512 (5)	C(1B)—C(5B)	1.503 (7)	
C(1A)—C(6A)	1.504 (6)	C(1B)—C(6B)	1.509 (4)	
C(2A)—C(3A)	1.445 (7)	C(2B)—C(3B)	1.456 (5)	
C(3A)—C(4A)	1.287 (7)	C(3B)—C(4B)	1.297 (5)	
C(6A)—C(7A)	1.519 (5)	C(6B)—C(7B)	1.523 (5)	
C(2A)—C(1A)—C(3A)	57.8 (3)	C(2B)—C(1B)—C(3B)	57.9 (2)	
C(2A)—C(1A)—C(5A)	117.5 (4)	C(2B)—C(1B)—C(5B)	117.8 (3)	
C(3A)—C(1A)—C(5A)	116.8 (3)	C(3B)—C(1B)—C(5B)	118.3 (3)	
C(2A)—C(1A)—C(6A)	117.8 (3)	C(2B)—C(1B)—C(6B)	116.7 (4)	
C(3A)—C(1A)—C(6A)	116.9 (4)	C(3B)—C(1B)—C(6B)	116.2 (3)	
C(5A)—C(1A)—C(6A)	116.9 (3)	C(5B)—C(1B)—C(6B)	116.9 (3)	
C(1A)—C(2A)—C(3A)	58.4 (3)	C(1B)—C(2B)—C(3B)	58.2 (2)	
C(1A)—C(3A)—C(2A)	63.8 (3)	C(1B)—C(3B)—C(2B)	63.9 (3)	
C(1A)—C(3A)—C(4A)	147.5 (4)	C(1B)—C(3B)—C(4B)	147.4 (4)	
C(2A)—C(4A)—C(4A)	148.6 (4)	C(2B)—C(3B)—C(4B)	148.7 (4)	
O(1A)—C(6A)—C(1A)	108.8 (3)	O(1B)—C(6B)—C(1B)	110.2 (3)	
O(1A)—C(6A)—C(7A)	108.8 (3)	O(1B)—C(6B)—C(7B)	106.2 (3)	
C(1A)—C(6A)—C(7A)	115.1 (4)	C(1B)—C(6B)—C(7B)	115.9 (3)	
D—H...A	D—H	H...A	D...A	D—H...A
(O1A)—H(1A)...O(1B)	0.77 (5)	1.98 (5)	2.782 (4)	171 (4)
(O1B)—H(1B)...O(1A')	0.81 (5)	2.01 (4)	2.835 (4)	157 (4)

Symmetry code: (i) 1 - x, -y, 2 - z.

Each crystal suffered from significant linear intensity decay which was corrected for accordingly. H atoms bonded to C atoms were included in the refinement in calculated positions (C—H 0.96 Å) and treated as riding atoms. The hydroxyl H atoms were refined with isotropic displacement parameters.

For both compounds, data collection: *CAD-4 Software* (Enraf-Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD-4 Software* (Siemens, 1993); program(s) used to solve structures: *SHELXTL/PC XS* (Sheldrick, 1990); program(s) used to refine structures: *SHELXTL/PC XLS3*; molecular graphics: *SHELXTL/PC XP*; software used to prepare material for publication: *SHELXTL/PC XPUBL*.

This research was supported by the Natural Science and Engineering Research Council (NSERC) Canada, the A. P. Sloan Foundation, the Merck Frosst Centre for Therapeutic Research, Eli Lilly Grantee Program, Bio-Mega and the University of Toronto.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1036). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Baldwin, J. E. & Widdison, W. C. (1992). *J. Am. Chem. Soc.* **114**, 2245–2251.
 Dewar, M. J. S. & Wasson, J. S. (1971). *J. Am. Chem. Soc.* **93**, 3081–3083.
 Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.

- Greenberg, A. & Liebman, J. F. (1978). In *Strained Organic Molecules*. New York: Academic Press.
 Lautens, M. & Delanghe, P. H. M. (1993). *J. Org. Chem.* **58**, 5037–5039.
 Lautens, M. & Delanghe, P. H. M. (1994). *J. Am. Chem. Soc.* **116**, 8526–8535.
 Ohta, T. & Takaya, H. (1991). In *Comprehensive Organic Synthesis*, edited by B. M. Trost. Oxford: Pergamon Press.
 Ramasubbu, N. & Venkatesan, K. (1982). *Acta Cryst.* **B38**, 976–978.
 Sheldrick, G. M. (1990). *SHELXTL/PC User's Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Siemens (1993). *XCAD-4 Software. Program to Extract Intensity Data from Enraf-Nonius CAD-4 Program*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1995). **C51**, 703–706

3,7-Dinitronoradamantane, C₉H₁₂N₂O₄, and 3,7,9-Trinitronoradamantane, C₉H₁₁N₃O₆

CLIFFORD GEORGE AND RICHARD D. GILARDI

Naval Research Laboratory, Washington DC 20375, USA

WALTER W. ZAJAC JR, JOHN H. BUZBY AND
 THOMAS R. WALTERS

*Department of Chemistry, Villanova University,
 Villanova, PA 19085-1699, USA*

(Received 2 March 1994; accepted 18 October 1994)

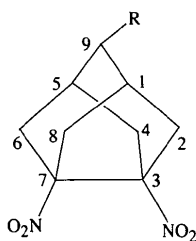
Abstract

The possible C_{2v} molecular symmetry for the dinitronoradamantane compound is not present; however, the carbon cages in each of the title compounds [3a,6a-dinitrooctahydro-2,5-methanopentalene and 3a,6a,7-trinitrooctahydro-2,5-methanopentalene, respectively] have approximate C_{2v} symmetry. Although the C—C—N—O torsion angles of the nitro groups differ significantly from the 90° required for C_{2v} molecular symmetry, approximate C_2 symmetry is maintained for both molecules in the asymmetric unit. Bond distances and angles are near expected values with the exception of the C3—C7 bond distance in both title compounds which averages 1.594 (4) Å.

Comment

Polynitropolycyclic cage molecules are of interest because of their possible use as high-energy density materials (Marchand, 1988) and polynitroadamantanes are compounds of this class for which there has been

considerable interest. There have been a number of structural determinations and syntheses of polynitroadamantanes reported including: 2,2-dinitroadamantane (Archibald & Baum, 1988; George & Gilardi, 1983), 1,3,5,7-tetranitroadamantane (Sollott & Gilbert, 1980; Zajac, Walters & Woods, 1989; George & Gilardi, 1984), 2,2,4,4-tetranitroadamantane (Dave, Ferraro, Ammon & Choi, 1990) and 2,2,6,6-tetranitroadamantane (Archibald & Baum, 1988), and 2,2,4,4,6,6-hexanitroadamantane (Dave, Bracuti, Axenrod & Liang, 1992). Noradamantane has about 85 kJ mol⁻¹ more strain than adamantane. For this reason, we have recently synthesized 3,7-dinitronoradamantane (Walters, Zajac & Woods, 1991; Klimova, Krayushkin, Sevost'yanova & Novikov, 1974), 3,7,9-trinitronoradamantane (Zajac, 1992), 3,7,7,9-tetranitronoradamantane (Zajac, 1993) and 2,2,6,6-tetranitronoradamantane (Zajac, Walters & Labroli, 1991), and determined the structures of dinitronoradamantane (1) and trinitronoradamantane (2) (Figs. 1 and 2, respectively). Structural studies for 3,7,9,9-tetranitronoradamantane (Gilardi, 1993) and 2,2,6,6-tetranitronoradamantane (Ammon, 1993) were of poor quality due to disorder or twinning.



- (1) R = H 3,7-Dinitronoradamantane
 (2) R = NO₂ 3,7,9-Trinitronoradamantane

The noradamantane cages in compounds (1) and (2) are almost identical (a least-squares fit of the C atoms has a mean deviation of 0.02 Å), both having approximate *mm*2 or *C*_{2v} symmetry. The bridged five-membered rings have envelope conformations and may be described by the ring torsion angles C_b—C_b—C—C, involving the bridge C atoms C3 and C7, whose absolute values average 32.4 (4)° with a maximum deviation from the average of 0.7°, and C—C_b—C_b—C, which average 0.4 (2)° with a maximum deviation from the mean of 0.3°. The dihedral angles between the rings, also given by the torsion angles C—C_b—C_b—C, average 113.9 (5)°. Compound (1) crystallized with two crystallographically distinct molecules in the asymmetric unit which differ only slightly, but most significantly, with respect to the orientation of the nitro groups. The nitro group torsion angles C_b—C_b—N—O involving the bridge C atoms are near ±60 or ±120° [deviations from these values are less than 4.0° except for C7—C3—N3—O3(A and B) = -68.8 (6) and 110.5 (6)°, respectively]. Compound (2) has a similar configuration

for the 3- and 7-nitro groups with torsion angles C_b—C_b—N—O deviating from ±60 or ±120° by between 1.0 and 7.3°. The orientation of the 9-nitro group may be given by C5—C9—N9—O9A = 33.6 (4)°. The rigid noradamantane-cage bond angles and distances for both compounds are near expected values except for C_b—C_b bond distances [1.598 (5) and 1.593 (5) Å in (1), and 1.591 (6) Å in (2)]. This is somewhat longer than the equivalent distance in the only other structural study of noradamantane reported, *i.e.* *N*-oxy-2-azanoradamantane (Dupeyre & Capiomont, 1977), where the equivalent C—C bond length is 1.574 (3) Å. A similar nitro-group substitution in bishomopentaprismanes has comparable values for the C2—C3 bond of 1.591 and 1.578 Å, respectively, for 2,3-dinitro- and 2,3,8-trinitrohexacyclo-[5.4.1.0^{2,6}.0^{3,10}.0^{4,8}.0^{9,10}]dodecane (Paquette, Fischer & Engel, 1985; Ammon & Paquette, 1991). Intermolecular distances correspond to normal van der Waals separations or greater.

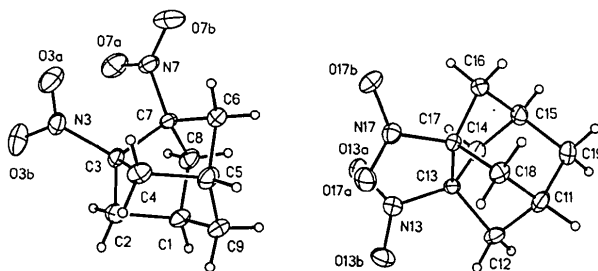


Fig. 1. A displacement ellipsoid plot of the asymmetric unit of (1) with ellipsoids drawn at the 20% probability level.

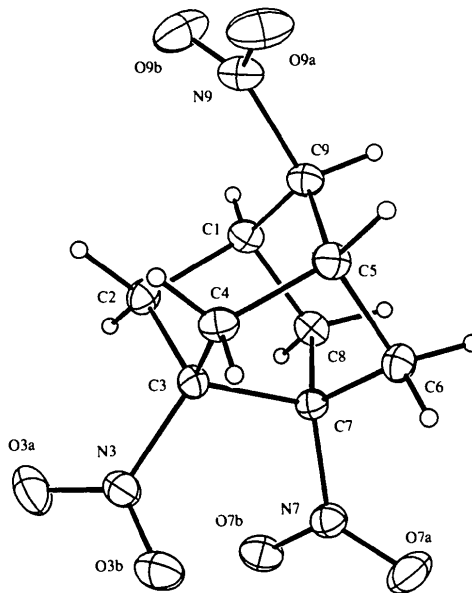


Fig. 2. A displacement ellipsoid plot of (2) with ellipsoids drawn at the 20% probability level.

Experimental

Compound (1)

Crystal data

C₉H₁₂N₂O₄

M_r = 212.2

Triclinic

P $\bar{1}$

a = 6.718 (2) Å

b = 12.270 (4) Å

c = 13.273 (4) Å

α = 64.19 (2)°

β = 81.76 (2)°

γ = 89.25 (2)°

V = 973.3 (5) Å³

Z = 4

D_x = 1.448 Mg m⁻³

Data collection

Siemens R3m/V diffractometer

2θ-θ scans

Absorption correction: none

3539 measured reflections

2563 independent reflections

1870 observed reflections

[*F* > 3σ(*F*)]

R_{int} = 0.016

Refinement

Refinement on *F*

R = 0.050

wR = 0.048

S = 1.68

1870 reflections

272 parameters

H-atom parameters not refined

w = 1/[σ²(*F*) + 0.00023*F*²]

(Δ/σ)_{max} = 0.06

Mo Kα radiation

λ = 0.71069 Å

Cell parameters from 25 reflections

θ = 9–13°

μ = 0.12 mm⁻¹

T = 295 K

Irregular

0.35 × 0.25 × 0.10 mm

Colorless

C12	0.1431 (6)	0.2151 (3)	-0.2993 (3)	0.050 (2)
C13	0.0355 (5)	0.2095 (3)	-0.1879 (3)	0.033 (1)
N13	0.1380 (5)	0.1371 (2)	-0.0870 (2)	0.043 (1)
O13A	0.0588 (4)	0.1308 (2)	0.0052 (2)	0.056 (1)
O13B	0.2914 (4)	0.0860 (2)	-0.0995 (2)	0.065 (1)
C14	-0.1830 (5)	0.1621 (3)	-0.1688 (3)	0.048 (2)
C15	-0.2720 (5)	0.2771 (3)	-0.2488 (3)	0.049 (2)
C16	-0.2089 (5)	0.3661 (3)	-0.2042 (3)	0.047 (2)
C17	0.0153 (5)	0.3475 (3)	-0.2110 (2)	0.033 (1)
N17	0.1173 (5)	0.3819 (2)	-0.1335 (2)	0.045 (1)
O17A	0.2971 (4)	0.3652 (2)	-0.1330 (2)	0.060 (1)
O17B	0.0176 (5)	0.4256 (3)	-0.0770 (2)	0.075 (2)
C18	0.1158 (5)	0.4184 (3)	-0.3343 (3)	0.046 (2)
C19	-0.1702 (6)	0.3172 (3)	-0.3719 (3)	0.058 (2)

Table 2. Selected geometric parameters (Å, °) for (1)

C1—C2	1.532 (7)	C1—C8	1.528 (5)
C1—C9	1.536 (4)	C2—C3	1.517 (5)
C3—N3	1.512 (6)	C3—C4	1.529 (4)
C3—C7	1.598 (5)	N3—O3A	1.215 (5)
N3—O3B	1.212 (4)	C4—C5	1.525 (6)
C5—C6	1.524 (5)	C5—C9	1.539 (6)
C6—C7	1.514 (4)	C7—N7	1.510 (5)
C7—C8	1.521 (6)	N7—O7A	1.211 (4)
N7—O7B	1.218 (5)	C11—C12	1.520 (5)
C11—C18	1.530 (6)	C11—C19	1.545 (5)
C12—C13	1.523 (5)	C13—N13	1.506 (4)
C13—C14	1.531 (5)	C13—C17	1.593 (5)
N13—O13A	1.232 (4)	N13—O13B	1.227 (4)
C14—C15	1.528 (4)	C15—C16	1.540 (6)
C15—C19	1.538 (5)	C16—C17	1.516 (5)
C17—N17	1.515 (5)	C17—C18	1.533 (4)
N17—O17A	1.223 (5)	N17—O17B	1.222 (5)
C2—C1—C8	99.8 (3)	C2—C1—C9	110.3 (3)
C8—C1—C9	110.1 (3)	C1—C2—C3	99.9 (3)
C2—C3—N3	114.5 (3)	C2—C3—C4	108.9 (3)
N3—C3—C4	112.0 (3)	C2—C3—C7	104.4 (3)
N3—C3—C7	112.8 (3)	C4—C3—C7	103.4 (3)
C3—N3—O3A	117.3 (3)	C3—N3—O3B	119.1 (4)
O3A—N3—O3B	123.6 (4)	C3—C4—C5	99.7 (3)
C4—C5—C6	99.7 (3)	C4—C5—C9	110.7 (3)
C6—C5—C9	110.6 (3)	C5—C6—C7	99.7 (3)
C3—C7—C6	104.6 (2)	C3—C7—N7	113.4 (3)
C6—C7—N7	114.2 (3)	C3—C7—C8	103.9 (3)
C6—C7—C8	108.8 (3)	N7—C7—C8	111.2 (3)
C7—N7—O7A	117.7 (3)	C7—N7—O7B	118.5 (3)
O7A—N7—O7B	123.8 (4)	C1—C8—C7	100.0 (3)
C1—C9—C5	110.9 (3)	C12—C11—C18	100.2 (3)
C12—C11—C19	111.1 (3)	C18—C11—C19	110.2 (3)
C11—C12—C13	99.4 (3)	C12—C13—N13	114.4 (3)
C12—C13—C14	109.1 (3)	N13—C13—C14	111.3 (2)
C12—C13—C17	104.7 (2)	N13—C13—C17	113.1 (3)
C14—C13—C17	103.5 (5)	C13—N13—O13A	117.1 (3)
C13—N13—O13B	119.4 (3)	O13A—N13—O13B	123.5 (3)
C13—C14—C15	99.7 (2)	C14—C15—C16	99.8 (3)
C14—C15—C19	110.8 (3)	C16—C15—C19	110.2 (3)
C15—C16—C17	99.3 (3)	C13—C17—C16	105.1 (3)
C13—C17—N17	113.8 (2)	C16—C17—N17	114.3 (3)
C13—C17—C18	103.6 (3)	C16—C17—C18	108.8 (3)
N17—C17—C18	110.5 (3)	C17—N17—O17A	117.0 (3)
C17—N17—O17B	118.8 (3)	O17A—N17—O17B	124.3 (4)
C11—C18—C17	99.3 (3)	C11—C19—C15	110.6 (3)

Compound (2)

Crystal data

C₉H₁₁N₃O₆

M_r = 257.2

Orthorhombic

*P*2₁*c**n*

a = 7.169 (1) Å

b = 10.875 (1) Å

c = 13.842 (2) Å

V = 1079.1 (3) Å³

Cu Kα radiation

λ = 1.5418 Å

Cell parameters from 25 reflections

θ = 25–32°

μ = 1.17 mm⁻¹

T = 295 K

Irregular

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

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
C1	0.4869 (6)	0.0793 (3)	0.2432 (3)	0.056 (2)
C2	0.5979 (6)	0.1114 (3)	0.3207 (3)	0.054 (2)
C3	0.5005 (5)	0.2282 (3)	0.3073 (3)	0.039 (2)
N3	0.5066 (5)	0.2598 (3)	0.4050 (3)	0.051 (2)
O3A	0.4516 (5)	0.3588 (3)	0.3933 (2)	0.083 (2)
O3B	0.5632 (5)	0.1859 (3)	0.4903 (2)	0.088 (2)
C4	0.5940 (6)	0.3308 (3)	0.1941 (3)	0.054 (2)
C5	0.4832 (6)	0.3036 (3)	0.1143 (3)	0.059 (2)
C6	0.2663 (6)	0.3022 (3)	0.1678 (3)	0.059 (2)
C7	0.2764 (5)	0.2098 (3)	0.2881 (3)	0.040 (2)
N7	0.1163 (5)	0.2196 (3)	0.3756 (3)	0.055 (2)
O7A	0.1118 (5)	0.1462 (3)	0.4733 (3)	0.087 (2)
O7B	-0.0004 (5)	0.3009 (3)	0.3445 (3)	0.095 (2)
C8	0.2692 (6)	0.0835 (3)	0.2935 (3)	0.057 (2)
C9	0.5323 (6)	0.1781 (3)	0.1206 (3)	0.062 (2)
C11	0.0609 (5)	0.3307 (3)	-0.3812 (3)	0.049 (2)

Z = 4	0.44 × 0.31 × 0.22 mm
D _x = 1.583 Mg m ⁻³	Colorless
Data collection	
Siemens R3m/V diffractometer	θ _{max} = 60.0°
2θ-θ scans	h = 0 → 8
Absorption correction: none	k = 0 → 12
1012 measured reflections	l = 0 → 15
878 independent reflections	3 standard reflections monitored every 97 reflections
831 observed reflections [F > 3σ(F)]	intensity decay: 0.022% (random)
R _{int} = 0.011	

Refinement

Refinement on F	Δρ _{max} = 0.14 e Å ⁻³
R = 0.032	Δρ _{min} = -0.11 e Å ⁻³
wR = 0.042	Extinction correction: empirical, isotropic; F = F[1 + 0.002χ ² /sin(2θ)] ^{-1/4}
S = 1.83	Extinction coefficient: χ = 0.041 (3)
831 reflections	Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
163 parameters	
Only coordinates of H atoms refined	
w = 1/[σ ² (F) + 0.00023F ²]	
(Δ/σ) _{max} = 0.03	

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (2)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U _{eq}
C1	1.0753 (7)	0.2878 (3)	0.4743 (2)	0.042 (1)
C2	1.1325 (7)	0.1537 (3)	0.4907 (2)	0.043 (1)
C3	1.1628 (7)	0.1141 (2)	0.3860 (2)	0.036 (1)
N3	1.1500 (7)	-0.0226 (2)	0.3699 (2)	0.048 (1)
O3A	1.1248 (7)	-0.0888 (2)	0.4391 (2)	0.078 (1)
O3B	1.1710 (7)	-0.0583 (2)	0.2876 (2)	0.066 (1)
C4	1.3501 (7)	0.1637 (3)	0.3517 (2)	0.041 (1)
C5	1.2992 (7)	0.2978 (3)	0.3304 (2)	0.043 (1)
C6	1.1297 (7)	0.2771 (3)	0.2634 (2)	0.045 (1)
C7	1.0151 (7)	0.1919 (3)	0.3258 (2)	0.036 (1)
N7	0.8735 (7)	0.1161 (2)	0.2719 (2)	0.045 (1)
O7A	0.8515 (7)	0.1338 (3)	0.1866 (2)	0.078 (1)
O7B	0.7855 (7)	0.0407 (2)	0.3187 (2)	0.062 (1)
C8	0.9159 (7)	0.2704 (3)	0.4032 (2)	0.044 (1)
C9	1.2291	0.3611 (3)	0.4228 (2)	0.042 (1)
N9	1.3917 (7)	0.3860 (3)	0.4914 (2)	0.052 (1)
O9A	1.5401 (7)	0.4099 (3)	0.4556 (2)	0.084 (1)
O9B	1.3618 (7)	0.3886 (3)	0.5770 (2)	0.085 (1)

Table 4. Selected geometric parameters (Å, °) for (2)

C1—C2	1.531 (5)	C1—C8	1.520 (6)
C1—C9	1.536 (5)	C2—C3	1.528 (4)
C3—N3	1.506 (4)	C3—C4	1.523 (7)
C3—C7	1.591 (6)	N3—O3A	1.212 (4)
N3—O3B	1.213 (4)	C4—C5	1.532 (5)
C5—C6	1.545 (6)	C5—C9	1.537 (4)
C6—C7	1.509 (5)	C7—N7	1.506 (5)
C7—C8	1.543 (5)	N7—O7A	1.207 (4)
N7—O7B	1.220 (5)	C9—N9	1.527 (5)
N9—O9A	1.202 (7)	N9—O9B	1.204 (4)
C2—C1—C8	100.3 (3)	C2—C1—C9	111.8 (4)
C8—C1—C9	107.7 (3)	C1—C2—C3	99.6 (2)

C2—C3—N3	114.2 (2)	C2—C3—C4	108.8 (3)
N3—C3—C4	110.9 (3)	C2—C3—C7	104.6 (3)
N3—C3—C7	114.1 (3)	C4—C3—C7	103.6 (3)
C3—N3—O3A	118.6 (3)	C3—N3—O3B	116.5 (3)
O3A—N3—O3B	124.8 (3)	C3—C4—C5	100.8 (3)
C4—C5—C6	99.5 (3)	C4—C5—C9	110.1 (2)
C6—C5—C9	107.9 (3)	C5—C6—C7	100.0 (3)
C3—C7—C6	105.3 (4)	C3—C7—N7	114.6 (3)
C6—C7—N7	114.8 (3)	C3—C7—C8	103.7 (2)
C6—C7—C8	108.0 (3)	N7—C7—C8	109.7 (4)
C7—N7—O7A	119.1 (3)	C7—N7—O7B	116.9 (3)
O7A—N7—O7B	124.0 (4)	C1—C8—C7	99.9 (4)
C1—C9—C5	112.9 (3)	C1—C9—N9	110.6 (3)
C5—C9—N9	110.3 (2)	C9—N9—O9A	117.2 (3)
C9—N9—O9B	118.7 (4)	O9A—N9—O9B	123.9 (5)

For both compounds, the data were corrected for Lorentz and polarization effects. H atoms were included using a riding model, where C—H = 0.96 Å, H angles were idealized, isotropic U values were fixed and coordinate shifts of bonded C atoms were applied to attached H atoms. For compound (2), the space group selection was based on E-value statistics and structure solution. The structures were solved by direct methods and refined by full-matrix least squares using the *SHELXTL-Plus* package (Sheldrick, 1989).

We (WWZ) are grateful for the financial support from Geo-Centers Inc. under the AMCCOM Contract DAAA21-89-C-0012. This work was supported by in part by the Office of Naval Research, Mechanics Division.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: CR1141). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Ammon, H. L. (1993). Unpublished results.
- Ammon, H. L. & Paquette, L. A. (1991). *Acta Cryst.* **C47**, 2159–2164.
- Archibald, T. G. & Baum, K. (1988). *J. Org. Chem.* **53**, 4645–4649.
- Dave, P. R., Bracuti, A., Axenrod, T. & Liang, B. (1992). *Tetrahedron*, **48**, 5839–5846.
- Dave, P. R., Ferraro, M., Ammon, H. L. & Choi, C. S. (1990). *J. Org. Chem.* **55**, 4461–4464.
- Dupeyre, R. M. & Capiomont, A. (1977). *J. Mol. Struct.* **42**, 243–250.
- George, C. & Gilardi, R. (1983). *Acta Cryst.* **C39**, 1674–1676.
- George, C. & Gilardi, R. (1984). *Acta Cryst.* **C40**, 674–676.
- Gilardi, R. (1993). Unpublished results.
- Klimova, T. A., Krayushkin, M. M., Sevost'yanova, V. V. & Novikov, S. S. (1974). *Izv. Akad. Nauk SSR Ser. Khim.* p. 2656.
- Marchand, A. P. (1988). *Tetrahedron*, **44**, 2377–2395.
- Paquette, L. A., Fischer, J. W. & Engel, P. (1985). *J. Org. Chem.* **50**, 2524–2527.
- Sheldrick, G. M. (1989). *SHELXTL-Plus*. Release 3.4 for Siemens R3m/V crystallographic system. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sollott, G. P. & Gilbert, E. E. (1980). *J. Org. Chem.* **45**, 5405–5408.
- Walters, T. R., Zajac, W. W. Jr & Woods, J. M. (1991). *J. Org. Chem.* **56**, 316–321.
- Zajac, W. W. Jr (1992). US Patent 5105031.
- Zajac, W. W. Jr (1993). US Patent 5189228.
- Zajac, W. W. Jr, Walters, T. R. & Labroli, M. A. (1991). Unpublished results.
- Zajac, W. W. Jr, Walters, T. R. & Woods, J. M. (1989). *J. Org. Chem.* **54**, 2468–2471.