made with *PARST* (Nardelli, 1983). Final positional and thermal parameters are given in Table 1.* Molecular geometry data are collected in Table 2. Fig. 1 shows the atomic numbering scheme.

Related literature. 7-Oxabicyclo[2.2.1]hept-5-en-2-one (7-oxanorbornenone) reacts with benzonitrile oxide to produce a mixture of regioisomers in a 65:35 ratio (Plumet, Escobar, Manzano, Arjona, Carrupt & Vogel, 1986). The regio- and stereochemistry of both adducts was tentatively assigned from the spectral data of the pure isomers, including NOE experiments. The present report describes the confirmation of the structure of the major isomer by X-ray diffraction.

We thank Professor J. Plumet, Organic Chemistry (Madrid), for providing us with the crystals and for helpful discussions.

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters, distances and angles involving H atoms, least-squares-planes' data and principal torsion angles have been deposited with British Library Document Supply Centre as Supplementary Publication No. SUP 52362 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1990). **C46**, 706–708

Structures of 1,3,5-Trinitro-2-oxo-1,3,5-triazacyclohexane (I) and 1,4-Dinitro-2,5-dioxo-1,4-diazacyclohexane (II)

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(*Received* 20 *June* 1989; *accepted* 24 *October* 1989)

Abstract. (I) $C_3H_4N_6O_7$, $M_r = 236{\cdot}10$, orthorhombic, *Pbnm* (non-standard setting of *Pnma*), $a = 5{\cdot}988$ (1), $b = 10{\cdot}052$ (2), $c = 13{\cdot}492$ (2) Å, $V = 812{\cdot}1$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}932$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 1{\cdot}63$ mm^{-1}, $F(000) = 480$, $T = 295$ K, final $R = 0{\cdot}034$, $wR = 0{\cdot}047$ for 516 observed reflections. (II) $C_4H_4N_4O_6$, $M_r = 208{\cdot}2$, orthorhombic, *C2cb* (non-standard setting of *Aba*2), $a = 6{\cdot}152$ (1), $b = 12{\cdot}961$ (2), $c = 9{\cdot}248$ (1) Å, $V = 737{\cdot}4$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}838$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 1{\cdot}50$ mm^{-1}, $F(000) = 416$, $T = 295$ K, final $R = 0{\cdot}037$, $wR = 0{\cdot}045$ for 538 observed reflections. Both compounds have only half a molecule per asymmetric unit; (I) lies on a mirror plane and (II) on a twofold rotation axis. In (I) the six-membered ring is a chair flattened at the keto end while in (II) it has a twisted conformation. The nitro groups are pyramidal. In (I) the out-of-plane tilt angles (between the C—N bond and the coincident C—N—C plane) for the nitro groups are 44·6 and 25·1°. In (II) this value is 22·2°. Close N···O intermolecular approaches occur in both molecules [2·94 Å in (I) and 2·82 Å in (II)].

Experimental. Both materials were synthesized by Clifford Coon of Lawrence Livermore Laboratory, Livermore, California. (I), colorless, 0·12 × 0·22 × 0·10 mm data crystal. Automated Nicolet *R*3*m* diffractometer with incident-beam graphite monochromator; 25 centered reflections within $30 \leq 2\theta \leq 76°$ used for determining cell parameters. Data corrected for Lorentz and polarization effects, but not for absorption. $2\theta_{\max} = 115°$; range of *hkl*: $0 \leq h \leq 6$, $-10 \leq k \leq 0$, $0 \leq l \leq 14$, standards, 204, $0\bar{4}0$, 004, monitored every 100 reflections with random variation of 3·0% over data collection, $\theta/2\theta$ mode, scan width $[2\theta(K\alpha_1) - 1{\cdot}0]$ to $[2\theta(K\alpha_2) + 1{\cdot}0]°$, scan rate a function of count rate (6° min^{-1} minimum, 30° min^{-1} maximum); 733 reflections measured, 586 unique, $R_{\rm int} = 0{\cdot}028$, 516 observed $[F_o > 3\sigma(F_o)]$. (II),

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^3$) *for molecule* (I)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O(1)	1975 (4)	3670 (2)	2500	41 (1)
N(2)	−58 (3)	2108 (2)	3362 (1)	31 (1)
N(4)	−2130 (4)	472 (2)	2500	31 (1)
O(2*a*)	1129 (4)	3739 (2)	4378 (1)	61 (1)
O(4*a*)	−5096 (3)	1330 (2)	3301 (1)	51 (1)
O(2*b*)	1154 (3)	1694 (2)	4890 (1)	54 (1)
C(3)	−864 (4)	704 (2)	3390 (2)	34 (1)
N(4*a*)	−4270 (4)	1114 (3)	2500	35 (1)
C(1)	784 (5)	2717 (3)	2500	30 (1)
N(2*a*)	839 (3)	2558 (2)	4279 (1)	40 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *for molecule* (I)

O(1)—C(1)	1·194 (4)	N(2)—C(3)	1·492 (3)
N(2)—C(1)	1·408 (2)	N(2)—N(2*a*)	1·422 (3)
N(4)—C(3)	1·439 (3)	N(4)—N(4*a*)	1·434 (4)
O(2*a*)—N(2*a*)	1·208 (3)	O(4*a*)—N(4*a*)	1·208 (2)
O(2*b*)—N(2*a*)	1·212 (3)		

C(3)—N(2)—C(1)	123·3 (2)	C(3)—N(2)—N(2*a*)	113·6 (2)
C(1)—N(2)—N(2*a*)	116·4 (2)	C(3)—N(4)—N(4*a*)	113·4 (1)
C(3)—N(4)—C(3*a*)	113·2 (3)	N(2)—C(3)—N(4)	107·6 (2)
N(4)—N(4*a*)—O(4*a*)	116·5 (1)	O(4*a*)—N(4*a*)—O(4*a*)	126·9 (3)
O(1)—C(1)—N(2)	124·3 (1)	N(2)—C(1)—N(2*a*)	111·4 (3)
N(2)—N(2*a*)—O(2*a*)	117·6 (2)	N(2)—N(2*a*)—O(2*b*)	115·0 (2)
O(2*a*)—N(2*a*)—O(2*b*)	127·3 (2)		

colorless, 0·10 × 0·28 × 0·30 mm data crystal. Automated Nicolet *R*3*m* diffractometer with incident-beam graphite monochromator; 25 centered reflections within $19 \le 2\theta \le 64°$ used for determining cell parameters. Data corrected for Lorentz and polarization effects, but not for absorption. $2\theta_{max} = 120°$; range of *hkl*: $0 \le h \le 6$, $0 \le k \le 14$, $0 \le l \le 9$, standards, 400, 040, 002, monitored every 100 reflections with random variation of 3·5% over data collection, $\theta/2\theta$ mode, scan width $[2\theta(K\alpha_1) - 1{\cdot}0]$ to $[2\theta(K\alpha_2) + 1{\cdot}0]°$, scan rate a function of count rate (8° min^{-1} minimum, 30° min^{-1} maximum); 992 reflections measured, 544 unique, $R_{int} = 0{\cdot}001$, 538 observed $[F_o > 3\sigma(F_o)]$.

Both structures solved by direct methods and refined using the full-matrix least-squares program provided with the MicroVAX version of the *SHELXTL* system (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o| + g(F_o|)^2]$. For (I), $g = 0{\cdot}00024$ and there were 88 parameters refined: atom coordinates for all atoms, anisotropic temperature factors for non-H atoms and isotropic temperature factors for H atoms; $(\Delta/\sigma)_{max} = 0{\cdot}01$, $R = 0{\cdot}034$, $wR = 0{\cdot}047$, $S = 1{\cdot}25$, final difference Fourier map excursions 0·18 and −0·19 e Å^{-3}. For (II), $g = 0{\cdot}00024$, secondary isotropic extinction from $F_c{}^* = F_c/[1{\cdot}0 + 0{\cdot}002(p)F_c{}^2/\sin 2\theta]^{0{\cdot}25}$ where $p =$

Table 3. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^3$) *for molecule* (II)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O(2*a*)	572	4846 (1)	2114 (2)	43 (1)
N(1)	727 (7)	4067 (1)	4359 (2)	31 (1)
O(1*a*)	262 (6)	2361 (1)	4526 (1)	45 (1)
N(1*a*)	1014 (7)	3057 (2)	3808 (2)	33 (1)
O(1*b*)	2029 (7)	2973 (2)	2693 (2)	45 (1)
C(2)	443 (6)	4893 (2)	3404 (2)	31 (1)
C(3)	−145 (9)	5880 (2)	4167 (3)	38 (1)

Table 4. *Bond lengths* (Å) *and bond angles* (°) *for molecule* (II)

N(1)—N(1*a*)	1·415 (3)	O(2*a*)—C(2)	1·197 (3)
N(1)—C(3*a*)	1·466 (4)	N(1)—C(2)	1·399 (3)
N(1*a*)—O(1*b*)	1·210 (4)	O(1*a*)—N(1*a*)	1·212 (3)
		C(2)—C(3)	1·506 (4)

N(1*a*)—N(1)—C(2)	119·7 (2)	N(1*a*)—N(1)—C(3*a*)	115·1 (2)
C(2)—N(1)—C(3*a*)	120·4 (2)	N(1)—N(1*a*)—O(1*a*)	116·3 (2)
N(1)—N(1*a*)—O(1*b*)	117·0 (2)	O(1*a*)—N(1*a*)—O(1*b*)	126·7 (2)
O(2*a*)—C(2)—N(1)	125·6 (2)	O(2*a*)—C(2)—C(3)	121·7 (2)
N(1)—C(2)—C(3)	112·6 (2)	C(2)—C(3)—N(1*a*)	112·8 (3)

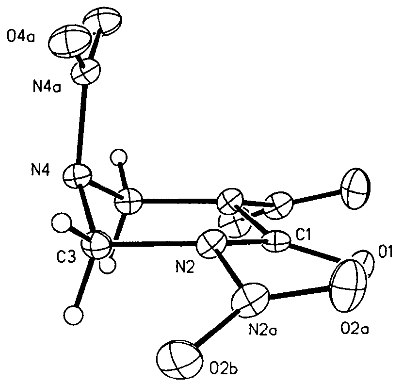

Fig. 1. Perspective drawing of the results of the X-ray study on the triazacyclohexane. Thermal ellipsoids are shown at the 20% probability level.

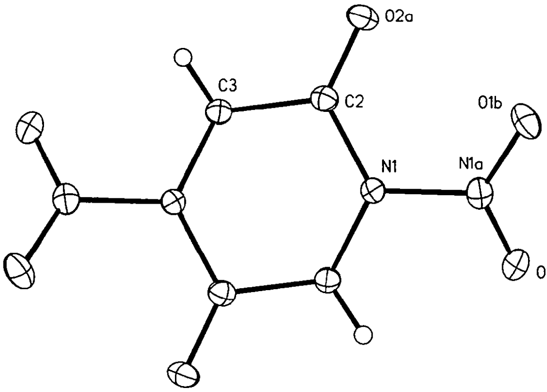

Fig. 2. Perspective drawing of the results of the X-ray study on the diazacyclohexane. Thermal ellipsoids are shown at the 20% probability level.

0·009 (1). There were 72 parameters refined: atom coordinates for all atoms, anisotropic temperature factors for non-H atoms and isotropic temperature factors for H atoms; $(\Delta/\sigma)_{max} = 0·01$, $R = 0·037$, $wR = 0·045$, $S = 2·23$, final difference Fourier map excursions 0·35 and $-0·37$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).* Atom numbering for Tables 1–4, atom coordinates, bond distances and bond angles for molecules (I) and (II) respectively, follows that shown in Figs. 1 and 2.

Related literature. Compound (I) is the monoketo analog of the commercial energetic compound RDX (Choi & Prince, 1972). It can also be compared to the structure of 1,3,5-tris(dimethylamino)hexahydro-1,3,5-triazine-2,4,6-trione (Zurn, Schwarz, Rozdzinski & Schmidt, 1982). A search of the current release of the Cambridge Structural Database did not produce any compounds similar to (II).

This work was supported in part by contract No. N00173-86-M-R828 from the ONR Mechanics Division.

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52363 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1990). C**46**, 708–711

Structures of Two Diastereomers of the Tetrahydrobenzothiophene Moiety of Breynolide

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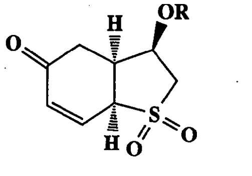
(*Received* 27 *July* 1989; *accepted* 26 *October* 1989)

Abstract. (1) (±)-3-Triisopropylsiloxy-2,3,3a,7a-tetrahydrobenzo[*b*]thiophen-5(4*H*)-one 1,1-dioxide, $C_{17}H_{30}O_4SSi$, $M_r = 358·57$, triclinic, $P\bar{1}$, $a = 7·4383(14)$, $b = 8·1000(15)$, $c = 16·895(3)$ Å, $\alpha = 80·202(14)$, $\beta = 88·800(15)$, $\gamma = 76·231(14)°$, $V = 974·0(3)$ Å^3, $Z = 2$, $D_x = 1·22$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·7107$ Å, $\mu = 2·339$ cm^{-1}, $F(000) = 388$, $T = 298$ K, $R = 0·0436$ for 3086 reflections $[F_o \geq 4\sigma(F_o)]$. (2) (±)-3-*tert*-Butyldiphenylsiloxy-2,3,3a,7a-tetrahydrobenzo[*b*]thiophen-5(4*H*)-one 1,1-dioxide, $C_{24}H_{28}O_4SSi$, $M_r = 440·63$, triclinic, $P\bar{1}$, $a = 9·113(2)$, $b = 11·020(2)$, $c = 12·551(3)$ Å, $\alpha = 74·01(2)$, $\beta = 70·83(2)$, $\gamma = 88·70(2)°$, $V = 1141·4(4)$ Å^3, $Z = 2$, $D_x = 1·28$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·7107$ Å, $\mu = 2·125$ cm^{-1}, $F(000) = 468$, $T = 298$ K, $R = 0·0424$ for 4262 reflections $[F_o \geq 4\sigma(F_o)]$. The two rings are *cis*-fused with the cyclohexenone ring in the sofa conformation and the tetrahydrothiophene ring in the half-chair conformation for both (1) and (2). However, the bulky siloxy groups assume different orientations in the two compounds. In (1), the triisopropylsiloxy group is equatorial and close intramolecular contacts between H2*A* and H4*B* [2·29 (3) Å], O11 and C4 [2·934 (3) Å] and O9 and C7 [2·979 (3) Å] are observed. In (2), the *tert*-butyldiphenylsiloxy group is axial and close contacts arise between O11 and C4 [2·864 (3) Å], O11 and C5 [2·930 (3) Å] and O9 and C7 [2·892 (3) Å].

Experimental. Compounds (1) and (2) were prepared *via* an intermolecular Diels–Alder reaction of *trans*-1-methoxy-3-trimethylsiloxy-1,3-butadiene (Danishefski & Kitahara, 1974) and the corresponding protected allylic alcohol obtained from 2,5-



(1) R = triisopropylsilyl
(2) R = *tert*-butyldiphenylsilyl

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