Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement coefficients* (Å^2 × 10^3)

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

	x	y	z	U_{eq}
C(1)	−374 (4)	1453 (3)	5791 (2)	32 (1)
O(1)	1375 (3)	1917 (2)	5471 (1)	47 (1)
N(2)	−1712 (3)	89 (3)	5508 (1)	37 (1)
N(2*a*)	−1380 (4)	−765 (3)	4660 (1)	48 (1)
O(2*a*)	−2833 (5)	−1787 (3)	4471 (2)	76 (1)
O(2*b*)	269 (4)	−458 (3)	4188 (1)	63 (1)
C(3)	−3857 (4)	−83 (3)	6017 (2)	41 (1)
C(4)	−3835 (4)	1405 (3)	6712 (2)	35 (1)
N(5)	−1592 (3)	2117 (3)	6560 (1)	37 (1)
N(5*a*)	−866 (3)	3460 (3)	7095 (2)	46 (1)
O(5*a*)	−2236 (3)	4058 (3)	7641 (1)	57 (1)
O(5*b*)	1079 (4)	3913 (3)	6996 (2)	91 (1)

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

C(1)—O(1)	1·185 (3)	C(1)—N(2)	1·396 (3)
C(1)—N(5)	1·396 (3)	N(2)—N(2*a*)	1·377 (3)
N(2)—C(3)	1·461 (3)	N(2*a*)—O(2*a*)	1·210 (3)
N(2*a*)—O(2*b*)	1·201 (3)	C(3)—C(4)	1·527 (4)
C(4)—N(5)	1·457 (3)	N(5)—N(5*a*)	1·368 (3)
N(5*a*)—O(5*a*)	1·210 (3)	N(5*a*)—O(5*b*)	1·212 (3)
O(1)—C(1)—N(2)	128·9 (2)	O(1)—C(1)—N(5)	128·5 (2)
N(2)—C(1)—N(5)	102·6 (2)	C(1)—N(2)—N(2*a*)	122·9 (2)
C(1)—N(2)—C(3)	115·3 (2)	N(2*a*)—N(2)—C(3)	119·7 (2)
N(2)—N(2*a*)—O(2*a*)	114·5 (2)	N(2)—N(2*a*)—O(2*b*)	119·2 (2)
O(2*a*)—N(2*a*)—O(2*b*)	126·3 (2)	N(2)—C(3)—C(4)	103·2 (2)
C(3)—C(4)—N(5)	102·4 (2)	C(1)—N(5)—C(4)	115·8 (2)
C(1)—N(5)—N(5*a*)	123·4 (2)	C(4)—N(5)—N(5*a*)	120·5 (2)
N(5)—N(5*a*)—O(5*a*)	116·0 (2)	N(5)—N(5*a*)—O(5*b*)	117·8 (2)
O(5*a*)—N(5*a*)—O(5*b*)	126·2 (3)		

Structure solved by direct methods. The least-squares refinement used the full-matrix program provided with the MicroVAX version of the *SHELXTL* system (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0{\cdot}00023$, secondary isotropic extinction from $F_c^* = F_c/[1{\cdot}0 + 0{\cdot}002(p)F_o^2/\sin 2\theta]^{0{\cdot}25}$ where $p = 0{\cdot}018$ (2). There were 126 parameters refined: atom coordinates for all atoms, anisotropic temperature factors for non-H atoms and isotropic temperature factors for H atoms; $(\Delta/\sigma)_{\max} = 0{\cdot}01$, $R = 0{\cdot}032$, $wR = 0{\cdot}045$, $S = 2{\cdot}09$, final difference Fourier excursions 0·27 and $-0{\cdot}21$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).† Atom numbering for Tables 1 and 2, atom coordinates, bond distances and bond angles, follows that shown in Fig. 1.

Related literature. The structure of 1,4-dinitroglycouril (DINGU) has been reported (Boileau, Wimmer, Gilardi, Stinecipher, Gallo & Pierrot, 1988).

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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 52317 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of the Product of Addition of 7-Oxabicyclo[2.2.1]hept-5-ene-2-carbonitrile and Benzonitrile Oxide

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Abstract. 5-Phenyl-3,10-dioxa-4-azatricyclo[5.2.1.$0^{2,6}$]dec-4-en-8-one, $C_{13}H_{11}NO_3$, $M_r = 229{\cdot}23$, orthorhombic, $Pna2_1$, $a = 5{\cdot}8737$ (4), $b = 17{\cdot}1877$ (12), $c = 10{\cdot}6692$ (10) Å, $V = 1077{\cdot}1$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}41$ g cm^{-3}. Mo $K\alpha$ radiation (graphite-crystal monochromator, $\lambda = 0{\cdot}71073$ Å), μ(Mo $K\alpha$) = 0·95 cm^{-1}, $F(000) = 480$, $T = 293$ K. Final conventional R factor = 0·025 for 688 unique reflections and

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197 variables. The structure was solved by direct methods. Distances and angles are normal. The angle between the least-squares plane through the phenyl ring and the five-membered ring is 10·9 (2)°. The C3—C2—C1, O21—C2—C1 and O21—C2—C3 angles are 105·5 (5), 126·5 (5) and 127·6 (5)° respectively. The C2—O21 bond distance is 1·219 (6) Å.

Experimental. Colorless crystal, 0·07 × 0·07 × 0·21 mm. Mo $K\alpha$ radiation was used with a graphite-crystal monochromator on a Nonius CAD-4 single-crystal diffractometer ($\lambda = 0{\cdot}71073$ Å). Unit-cell dimensions were from the angular settings of 25 reflections with $20 < \theta < 30°$. Space group $Pna2_1$ from the systematic absences. 7658 reflections were measured, *hkl* range (−5, −24, −10) to (8, 24, 15), theta limits ($0 < \theta < 30°$). ω–2θ scan technique and a variable scan rate with a maximum scan time of 60 s per reflection. Intensity was checked by monitoring three standard reflections every 60 min. Final drift correction factors were between 0·99 and 1·02. On all reflections profile analysis was performed (Lehmann & Larsen, 1974; Grant & Gabe, 1978); an empirical absorption correction was applied, using ψ scans (North, Phillips & Mathews, 1968), $\mu(\text{Mo } K\alpha) = 0{\cdot}95\ \text{cm}^{-1}$ (correction factors in the range 0·97 to 1·00). Symmetry equivalent reflections averaged, $R_{\text{int}} = \sum(I - \langle I\rangle)/\sum I = 0{\cdot}046$, resulting in 2121 unique reflections of which only 688 were observed with $I > 3\sigma(I)$. Lorentz and polarization corrections were applied and the data reduced to $|F_o|$ values. The structure was solved by directs methods, using the program *SHELXS*86 (Sheldrick, 1985).

Isotropic least-squares refinement, using *SHELX* (Sheldrick, 1976), converged to $R = 0{\cdot}068$. At this stage an additional empirical absorption correction was applied (Walker & Stuart, 1983). Maximum and minimum absorption correction factors were 1·07 and 0·44 respectively. Further anisotropic refinements followed by a difference Fourier synthesis allowed the location of all the hydrogen atoms.

During the final stages of the refinement the positional parameters and the anisotropic thermal parameters of the non-hydrogen atoms were refined. All hydrogens were refined isotropically. The final conventional agreement factors were $R = 0{\cdot}025$ and $wR = 0{\cdot}023$ for the 688 'observed' reflections and 197 variables. The function minimized was $\sum w(F_o - F_c)^2$, $w = 1/[\sigma^2(F_o) + 0{\cdot}00020F_o^2]$ with $\sigma(F_o)$ from counting statistics. The maximum shift over e.s.d. ratio in the last full-matrix least-squares cycle was less than 0·04. The final difference Fourier map showed no peaks higher than 0·075 e Å^{-3} and deeper than −0·070 e Å^{-3}. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The plot was made with program *PLUTO* (Motherwell, 1976). Geometrical calculations were

Table 1. *Fractional positional and thermal* (Å^2 × 10^2) *parameters, with e.s.d.'s in parentheses*

$$U_{\text{eq}} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0·310 (1)	0·1635 (3)	0·7771 (7)	3·9 (2)
C2	0·355 (1)	0·1641 (3)	0·9171 (6)	4·2 (2)
O21	0·5287 (8)	0·1853 (2)	0·9692 (5)	5·7 (1)
C3	0·149 (1)	0·1249 (4)	0·9761 (7)	4·8 (2)
C4	0·026 (1)	0·1008 (3)	0·8573 (6)	4·1 (2)
O41	0·064 (6)	0·1663 (2)	0·7752	4·5 (1)
C5	0·154 (9)	0·0363 (3)	0·7916 (6)	3·6 (2)
O51	0·032 (6)	0·0051 (2)	0·6850 (5)	5·3 (1)
C6	0·358 (1)	0·0804 (3)	0·7344 (7)	3·7 (2)
N61	0·137 (8)	0·0297 (3)	0·5727 (6)	5·0 (2)
C6′	0·316 (1)	0·0693 (3)	0·5972 (5)	3·6 (2)
C61	0·4536 (9)	0·1038 (3)	0·4977 (6)	3·5 (2)
C62	0·378 (1)	0·1028 (4)	0·3731 (7)	4·8 (3)
C63	0·511 (1)	0·1378 (3)	0·2816 (7)	5·8 (3)
C64	0·716 (1)	0·1733 (4)	0·3093 (7)	5·6 (3)
C65	0·790 (1)	0·1746 (3)	0·4326 (7)	5·1 (2)
C66	0·660 (1)	0·1387 (3)	0·5242 (7)	4·1 (2)

Table 2. *Bond lengths* (Å) *and bond angles* (°), *with e.s.d.'s in parentheses*

C1—C2	1·517 (8)	O51—N61	1·412 (5)
C1—O41	1·446 (6)	C6—C6′	1·496 (7)
C1—C6	1·525 (7)	N61—C6′	1·280 (6)
C2—O21	1·219 (6)	C6′—C61	1·460 (7)
C2—C3	1·519 (8)	C61—C62	1·401 (7)
C3—C4	1·517 (7)	C61—C66	1·382 (7)
C4—O41	1·445 (5)	C62—C63	1·388 (7)
C4—C5	1·513 (7)	C63—C64	1·383 (8)
C5—O51	1·446 (5)	C64—C65	1·385 (8)
C5—C6	1·544 (6)	C65—C66	1·386 (8)
C2—C1—O41	100·7 (5)	C1—C6—C5	101·5 (4)
C2—C1—C6	105·6 (4)	C1—C6—C6′	112·5 (5)
O41—C1—C6	102·1 (4)	C5—C6—C6′	101·4 (5)
C1—C2—O21	126·5 (5)	O51—N61—C6′	110·2 (4)
C1—C2—C3	105·5 (5)	C6—C6′—N61	113·7 (6)
O21—C2—C3	127·6 (5)	C6—C6′—C61	124·7 (5)
C2—C3—C4	98·9 (5)	N61—C6′—C61	121·4 (5)
C3—C4—O41	102·7 (4)	C6′—C61—C62	120·7 (5)
C3—C4—C5	110·4 (5)	C6′—C61—C66	120·8 (5)
O41—C4—C5	102·3 (4)	C62—C61—C66	118·5 (6)
C1—O41—C4	97·0 (4)	C61—C62—C63	118·9 (6)
C4—C5—O51	112·9 (4)	C62—C63—C64	122·1 (6)
C4—C5—C6	102·1 (4)	C63—C64—C65	118·9 (7)
O51—C5—C6	104·7 (4)	C64—C65—C66	119·2 (6)
C5—O51—N61	109·9 (4)	C61—C66—C65	122·2 (6)

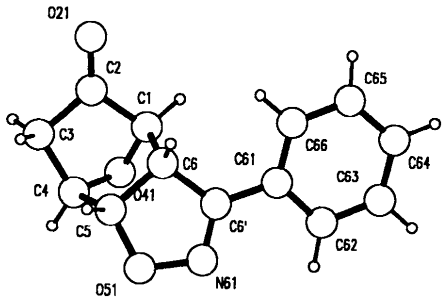

Fig. 1. *PLUTO* (Motherwell, 1976) diagram showing the molecular geometry and the atomic numbering.

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.

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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.

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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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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}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ 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}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ 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, $0{\cdot}12 \times 0{\cdot}22 \times 0{\cdot}10$ mm data crystal. Automated Nicolet *R*3*m* diffractometer with incident-beam graphite monochromator; 25 centered reflections within $30 \le 2\theta \le 76°$ used for determining cell parameters. Data corrected for Lorentz and polarization effects, but not for absorption. $2\theta_{\max} = 115°$; range of *hkl*: $0 \le h \le 6$, $-10 \le k \le 0$, $0 \le l \le 14$, standards, 204, $0\overline{4}0$, 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, $R_{\mathrm{int}} = 0{\cdot}028$, 516 observed $[F_o > 3\sigma(F_o)]$. (II),