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6,6,8-Trinitropentacyclo[5.3.0.$0^{2,5}$.$0^{3,9}$.$0^{4,8}$]decane, $C_{10}H_9N_3O_6$

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Abstract. $M_r = 267{\cdot}2$, monoclinic, $P2_1/c$, $a = 9{\cdot}352\ (4)$, $b = 7{\cdot}401\ (2)$, $c = 16{\cdot}070\ (6)$ Å, $\beta = 97{\cdot}76\ (4)°$, $V = 1102{\cdot}1$ Å^3, $Z = 4$, $D_x = 1{\cdot}61$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 1{\cdot}19$ mm^{-1}, $F(000) = 552$, $T = 292$ K, $R = 0{\cdot}059$, $R_w = 0{\cdot}051$ for 1495 observed reflections. The molecule is substantially distorted from a cubane geometry; two of the six sides of the framework are four-membered rings and four sides are five-membered rings. The average C—C—C angles in the four- and five-membered rings are 89·2 and 102·9°, respectively. C—C bond lengths show the effects of the nitro substituents: the average of the two bonds linked to $C(NO_2)_2$ is 1·507 (4) Å, and the three bonds joined to $C(NO_2)$ average 1·560 (4) Å.

Introduction. There is considerable interest in the synthesis and chemistry of strained energetic compounds (*e.g.* Sollott & Gilbert, 1980; Eaton, Ravi Shankar, Price, Pluth, Gilbert, Alster & Sandus, 1984; Marchand & Suri, 1984). Polynitropolycyclic compounds are potential members of this important class. In this paper, we report the structure of the first polynitrobishomocubane (I) to have been prepared (Marchand & Suri, 1984). The structure of one other homocubane (homocubanecarboxylic acid *p*-bromoanilide; Pettersen, 1967) has been reported; these structural data, however, are not readily available.

Experimental. Six-sided parallelepiped crystal, 0·49 × 0·23 × 0·19 mm, from ethyl acetate–hexane; cell parameters determined and intensity measurements made with a Picker FACS-I diffractometer, graphite monochromator, Cu radiation; unit-cell parameters determined by least squares from 2θ values of 14 reflections automatically centered at $\pm 2\theta$; intensities measured with θ–2θ scan technique, 2° min^{-1} 2θ scan speed, 10 s backgrounds, 2θ scan width = 1·6° + 0·3° tanθ; four standards monitored at 100 reflection intervals, maximum intensity variation <2%; 2θ maximum of 126°, *h,k,l* range 0–10, 0–8, −18–18; 2111 total reflections measured, 1901 unique (excluding systematic absences), 1495 3σ above background; Gaussian quadrature (8 × 8 × 8 grid) absorption correction ($A^*_{\max}/A^*_{\min} = 1{\cdot}558/1{\cdot}234$); structure solved with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); H atoms located in difference map; full-matrix least-squares structure refinement minimized $\sum w(F_o - F_c)^2$, $w = 1/\sigma^2(F)$; C, N, and O atoms refined with anisotropic

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temperature factors, H atoms with isotropic terms; maximum shift/error ratio of 0·21 in final least-squares cycle; final R and R_w of 0·059 and 0·051; C, N, and O scattering factors from Cromer & Mann (1968), H from Stewart, Davidson & Simpson (1965); maximum and minimum peak heights in final difference map of 0·26 and $-0·21$ e Å^{-3}. All calculations were carried out at the University of Maryland's Computer Science Center on a Univac 1100/82 computer with *MULTAN*80 and the *XRAY* system (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976).

Discussion. Atomic coordinates and temperature factors for C, N, O, and H are listed in Table 1;[*] an *ORTEP* (Johnson, 1971) drawing is shown in Fig. 1.

The C—C bond lengths fall into two groups: the four bonds involved with bridging atoms C(9) and C(10) range from 1·505 (4) to 1·525 (4) Å and average 1·513 (4) Å; the cubane-skeleton bonds range from 1·526 (5) to 1·570 (4) Å and average 1·550 (4) Å. In cubane (Fleischer, 1964) and 1,4-dinitrocubane (Eaton *et al.*, 1984) the average C—C distances are 1·551 (3) and 1·558 (3) Å, respectively. The C—C—C bond angles in the two four-membered rings range from 85·6 (2) to 92·8 (2)° and average 89·2 (2)°. In the four five-membered rings, the endocyclic angles opposite the bridging atoms C(9) and C(10) average 102·9 (2)°; the enlargement reflects the distortion of the cubane skeleton at C(1)···C(2) and C(3)···C(8) by the bridging atoms. The C—C—C angles at C(9) and C(10) are 98·4 (2) and 95·7 (3)°, respectively. Four of the faces of the original cubane skeleton are bent about their diagonals by approximately 20° (Table 2), whereas the other two cubane faces are to be essentially planar and almost parallel to each other. The two C—NO_2 groups attached to C(9) are both essentially planar, and the largest deviations of atoms from each mean plane are 0·016 Å for N(1) group and 0·010 Å for N(2) groups. The torsional angles of the mean planes measured from the common plane, N(1)—C(9)—N(2), about each C(9)—N axis are 17·2° and 73·0° for N(1) and N(2) groups, respectively, as shown in Table 2. The relative torsional angle between the two C—NO_2 groups therefore is 89·8°. The C(10)—NO_2 group is slightly bent with a largest atomic positional deviation from the mean plane of 0·17 Å.

The C(7)—NO_2 distance of 1·488 (4) Å is fairly typical of C(sp^3)—NO_2 bonds where the C atom bears a single electron-withdrawing substituent (the NO_2 group). The C—N length in 1,4-dinitrocubane (Eaton *et al.*, 1984) is 1·466 (2) Å. Additionally, the C(9)—N(1)

Table 1. *Fractional coordinates and equivalent isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ where a_i are the unit-cell axes and U_{ij} are the anisotropic temperature factors in the form $\exp(-2\pi^2\sum_i\sum_j U_{ij}h_ih_ja_i^*a_j^*)$.

	x	y	z	$U_{eq}(\times 10)$
C(1)	0·1401 (3)	0·2679 (4)	0·2836 (2)	0·53 (2)
C(2)	0·3409 (3)	0·1639 (4)	0·3661 (2)	0·43 (3)
C(3)	0·3443 (4)	0·3679 (4)	0·3882 (2)	0·38 (4)
C(4)	0·1908 (4)	0·4220 (5)	0·3467 (2)	0·63 (4)
C(5)	0·0919 (4)	0·3520 (5)	0·4094 (2)	0·73 (5)
C(6)	0·0759 (3)	0·1763 (4)	0·3551 (2)	0·52 (5)
C(7)	0·2109 (3)	0·1087 (4)	0·4131 (2)	0·46 (5)
C(8)	0·1985 (3)	0·2629 (4)	0·4781 (2)	0·52 (6)
C(9)	0·2767 (3)	0·1666 (4)	0·2748 (2)	0·38 (6)
C(10)	0·3380 (4)	0·3686 (6)	0·4825 (2)	0·44 (7)
N(1)	0·2485 (3)	−0·0150 (4)	0·2325 (2)	0·46 (7)
N(2)	0·3736 (4)	0·2632 (5)	0·2199 (2)	0·52 (7)
N(3)	0·2140 (4)	−0·0789 (4)	0·4466 (2)	0·82 (7)
O(1)	0·2004 (2)	−0·1313 (3)	0·2736 (2)	0·67 (8)
O(2)	0·2684 (3)	−0·0331 (4)	0·1605 (2)	0·80 (8)
O(3)	0·4972 (3)	0·2117 (5)	0·2260 (2)	1·07 (8)
O(4)	0·3220 (3)	0·3855 (4)	0·1765 (2)	1·13 (9)
O(5)	0·1025 (3)	−0·1578 (4)	0·4451 (2)	1·34 (9)
O(6)	0·3313 (3)	−0·1399 (4)	0·4768 (2)	1·4319
H(1)	0·071 (4)	0·296 (5)	0·230 (2)	0·8 (1)
H(2)	0·419 (3)	0·103 (4)	0·383 (2)	0·47 (9)
H(3)	0·418 (3)	0·424 (4)	0·370 (2)	0·38 (8)
H(4)	0·182 (3)	0·530 (4)	0·332 (2)	0·5 (1)
H(5)	0·019 (3)	0·417 (4)	0·425 (2)	0·6 (1)
H(6)	−0·016 (4)	0·079 (5)	0·352 (2)	0·8 (1)
H(7)	0·164 (3)	0·230 (4)	0·523 (2)	0·45 (8)
H(8)	0·419 (3)	0·302 (4)	0·512 (2)	0·6 (1)
H(9)	0·325 (4)	0·502 (6)	0·502 (2)	0·9 (1)

Table 2. *Torsional angles to show the degree of bending for each of the six faces of the bishomocubane skeleton*

The torsional angles of the mean planes of the two C(9)—NO_2 groups, with respect to the plane defined by the two C(9)—N bonds, are also given.

Plane 1	Plane 2	Axis	Torsion (°)
C(1), C(5), C(6)	C(1), C(5), C(4)	C(1)—C(5)	19·8 (3)
C(5), C(7), C(6)	C(5), C(7), C(8)	C(5)—C(7)	18·8 (3)
C(2), C(4), C(1)	C(2), C(4), C(3)	C(2)—C(4)	18·8 (3)
C(3), C(7), C(2)	C(3), C(7), C(8)	C(3)—C(7)	20·7 (3)
C(3), C(5), C(4)	C(3), C(5), C(8)	C(3)—C(5)	1·3 (4)
C(1), C(7), C(2)	C(1), C(7), C(6)	C(1)—C(7)	2·1 (2)
C(9), N(1), O(1), O(2)	N(1), C(9), N(2)	N(1)—C(9)	17·2 (3)
C(9), N(2), O(3), O(4)	N(1), C(9), N(2)	N(2)—C(9)	73·0 (3)

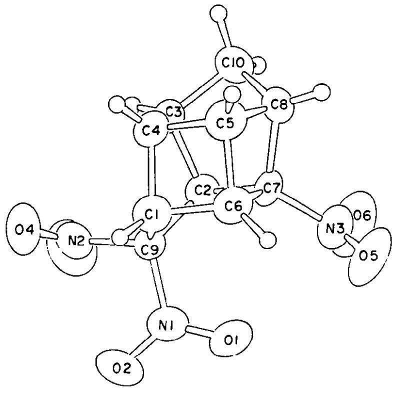

Fig. 1. *ORTEP* drawing (Johnson, 1971) with the thermal ellipsoids at 50% probability.

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[*] Tables containing structure factors, anisotropic temperature factors and bond lengths and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39825 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

and C(9)—N(2) lengths of 1·513 (4) and 1·525 (4) Å reflect the effect of two or more electron-withdrawing groups on the same C atom. In *N,N'*-bis(2,2,2-trinitroethyl)oxamide (Bhattacharjee & Ammon, 1982), for example, a compound containing a C(NO$_2$)$_3$ group, the average C—N distance is 1·523 (4) Å. Each nitro group is undergoing a strong librational motion about the C—N bond or the central N atom, as evidenced by the large thermal ellipsoids of the O atoms. Consequently, all observed N—O bond lengths are shortened by unknown amounts.

There are no unusual intermolecular contacts.

There is current interest in the prediction of the crystal densities of energetic materials. The values for (I) calculated with the empirical methods of Cady (1979) and Stine (1981) are 1·67 and 1·69 Mg m^{-3}, respectively. The method of Cady calculates a molecular volume by summing the appropriate atom and group-volume increments and includes an empirically derived packing coefficient (unit-cell volume/total molecular volume) to give an effective molecular volume in the crystal, while that of Stine calculates an effective crystal molecular volume from atom and group-volume increments derived from a large structure-density calibration base. A value of 1·63 Mg m^{-3} was obtained with a very simple weight fraction of hydrogen *vs* crystal-density relationship developed several years ago (Ammon, 1980) for use with energetic compounds. The predicted values are in reasonable agreement with the X-ray density of 1·61 Mg m^{-3}, considering that none of the methods was calibrated with cage molecules. Packing calculations, based on the atomic radii listed by Kitaigorodsky (1973), have been carried out for more than 50 C, H, N, O and F containing polynitroorganic compounds (Holden, 1984). The packing coefficients (PC = total molecular volume/unit-cell volume) range from 0·70 in [FC(NO$_2$)$_2$CH$_2$C(=NH)]$_2$ to 0·81 in 2,4,6-trinitro-1,3,5-benzenetriamine. The PC of 0·72 calculated for (I) is at the lower end of the range, suggesting that the compound's crystal density is due, at least in part, to inefficient crystal packing.

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Structure of 7,12-Diphenyl-4a,12-dihydroindeno[7a,1-*b*]naphthalene, $C_{29}H_{22}$

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Abstract. $M_r = 370\cdot51$, orthorhombic, *Pbca*, $a = 9\cdot968\ (3)$, $b = 17\cdot163\ (1)$, $c = 23\cdot583\ (2)$ Å, $U = 4034\cdot59$ Å^3, $Z = 8$, $D_x = 1\cdot22$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0\cdot529$ cm^{-1}, $F(000) = 1568\cdot00$, $T = 295$ K, $R = 0\cdot037$ for 2304 observed reflections. The structure consists of a roughly planar, partly hydro-

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