355 Å^3 at 100% RH using areas per lipid of 59.3 and 65.5 Å^2, respectively. Terminal methyl-group volumes increased with increased levels of humidity from 51 (1) at 70% RH to 56 (2) Å^3 at 100% RH.

In conclusion, we have clearly demonstrated, using one-dimensional absolute electron-density profiles calculated by means of direct-phasing methods, quantitative structural differences between two phases of DLPC. In addition to the changes observed by other researchers when comparing gel and liquid-crystalline bilayers, our profiles clearly show the phosphorylcholine headgroup reorienting when the DLPC bilayers undergo a transition from the 3D-L_δ phase to the L_α phase. Although a 2D-L_δ phase has been previously observed (Tardieu *et al.*, 1973), we have reported and quantified the physical characteristics for the first time of a DLPC 3D-L_δ phase which seems to differ substantially from the two-dimensional phase.

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Structure of 1,1,5,5-Tetranitro-[4]peristylane. Structure Solution from Molecular Packing Analysis

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Abstract

The structure of the title compound, decahydro-2,2,5,5-tetranitro-1,6:3,4-dimethanocyclobuta[1,2:3,-4]dicyclopentene, $C_{12}H_{12}N_4O_8$, was solved with the molecular packing program *MOLPAK*, starting with an *AM*1-geometry-optimized model of an isolated molecule. The 20 best predicted crystal structures

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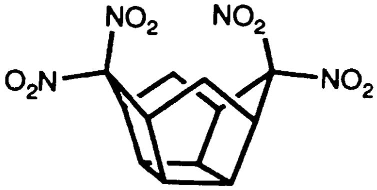
from the *MOLPAK* procedure were subjected to lattice energy refinement with the *WMIN* program. A structure-factor calculation with the top *MOLPAK*/*WMIN* derived structure and the 197 F_o data to $\sin\theta/\lambda = 0.324$ Å^{-1} ($\theta = 30°$) gave an R value of 0.29. Final $R = 0.057$, $wR = 0.067$ for 730 reflections with $I > 3\sigma(I)$. There are no unusual intermolecular distances and crystal packing is normal.

Introduction

One of the most important properties of an energetic material is the solid-state density. The performance characteristics of propellants and explosives, such as detonation velocity and pressure, are functions of density and at an even more basic level density determines the quantity of material that can be packed into a limited volume. Polynitro-cage compounds, such as the cubanes and homocubanes, are of interest as potential high-energy density materials. The energy contained in the strained ring systems together with their rather compact shapes holds the promise of energetic materials with exceptional performance characteristics.

We have undertaken crystal structure determinations of several of the [4]peristylanes and bishomopentaprismanes to enable detailed crystal-packing studies to be made and to confirm, in some cases, reported crystal densities. In this paper we report the structure of (I). The solution was achieved by an *ab initio* molecular packing procedure that has been under development in our laboratory over the last several years; this is described in some detail.



(I)

Experimental

Experimental details are given in Table 1. Data were collected with Enraf–Nonius diffractometer software (Enraf–Nonius, 1989). The θ-scan width was $1.5(0.8 + 0.14\tan\theta)°$, recorded as 96 steps with two outermost 16-step blocks for background determination; the θ-scan speed was 2.24–0.82° min^{-1}. Refinement was by full-matrix least squares, minimizing $\sum[1/\sigma^2(F_o)(F_o - F_c)^2]$ with anisotropic temperature factors for C, O and N; H-atom positions were obtained from the C-atom framework, with individual isotropic temperature factors refined.*

Table 1. *Experimental details*

Crystal data	
Chemical formula	$C_{12}H_{12}N_4O_8$
Molecular weight	340.3
Crystal system	Monoclinic
Space group	$P2_1$
a (Å)	7.073 (1)
b (Å)	11.624 (1)
c (Å)	8.231 (1)
β (°)	96.79 (1)
V (Å^3)	671.7 (8)
Z	2
Radiation type	Cu $K\alpha$
Wavelength (Å)	1.54178
No. of reflections for cell parameters	25
θ range for cell parameters (°)	7.4-25.2
μ (mm^{-1})	1.20
Temperature (K)	293
Crystal color	Colorless
Crystal form	Rod
Crystal size (mm)	0.40 × 0.037 × 0.033
D_x (Mg m^{-3})	1.682
Crystal source	Supplied by L. A. Paquette
Data collection	
Diffractometer	Enraf-Nonius CAD-4
Absorption correction	None
No. of measured reflections	1285
No. of independent reflections	1054
No. of observed reflections	730
Observation criterion	$[I > 3\sigma(I)]$
$R_{\rm int}$	0.030 for 78 pairs
$\theta_{\rm max}$ (°)	60.0
Range of h, k, l	$-7 \rightarrow h \rightarrow 7$ $-11 \rightarrow k \rightarrow 0$ $0 \rightarrow l \rightarrow 8$
No. of standard reflections	7
Frequency of standard reflections	60 min of X-ray exposure
Intensity variation (%)	$-2.8 \rightarrow 0.7\%$, $\langle -1.8\% \rangle$
Refinement	
Refinement on	F
Final R factor	0.057
wR	0.067
S	1.7
No. of reflections used in refinement	730
No. of parameters used	228
H-atom refinement	Only H-atom U's refined
Weighting	$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{\rm max}$	0.09
$\Delta\rho_{\rm max}$ (e Å^{-3})	0.28
$\Delta\rho_{\rm min}$ (e Å^{-3})	-0.31
Source of atomic scattering factors	*International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 155-175)

All crystallographic calculations were performed with the *TEXSAN* (Molecular Structure Corporation, 1989) program system on DEC MicroVAX II, VAXStation II and 3100 computers; structure solution was performed with the molecular packing program *MOLPAK* (Holden, Du & Ammon, 1993). Figs. 1 and 2 were drawn using the *Chem*3D pro-

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71570 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1059]

gram (Cambridge Scientific Computing, 1990) on an Apple MacIntosh IIx workstation and printed on an Apple LaserWriter IINTX. Figs. 3 and 4 were drawn with the *ORTEP* (Johnson, 1976) subprogram of *TEXSAN*; the *PLOTMD* program (Luo, Ammon & Gilliland, 1989) was used to display the drawings on a VAXStation monitor, add labels and prepare print files for a Hewlett–Packard Laser-Jet II printer.

The procedure used to predict possible crystal structures has been previously described in detail (Holden, Du & Ammon, 1993). A brief outline will be given here. Investigations of the molecular coordination environments in a number of triclinic–orthorhombic crystal structures with $Z \leq 4$ and one molecule per asymmetric unit have revealed common patterns in the coordination geometries for each of the space groups. The most common coordination for $P2_1$ is illustrated in Fig. 1. The molecular packing program *MOLPAK* was written to reconstruct the various geometries, starting with models for the compounds of interest. *MOLPAK* uses a rigid-body model of the compound (the search probe) to build a number of packing arrangements (possible crystal structures) with the predetermined coordination geometry. All unique orientations of the search probe (with respect to fixed Cartesian axes) were examined and the molecules were fit together (in the appropriate coordination geometry) with a repulsion-only potential function. The best (smallest volume) of the *MOLPAK* packing arrangements are refined with the *WMIN* procedure (Busing, 1981).

For this investigation, the search probe structure was constructed with the *Macromodel* (Still, 1990) system and subjected to geometry optimization with the *AM*1 procedure (Dewar, Zoebisch, Healy & Stewart, 1985; Stewart, 1990). The probe and refined X-ray structures, which are shown in Fig. 2, differ primarily in C—NO_2 conformations. In the former, the pairs of geminal nitro substituents are arranged with the upper and lower nitro O atoms approximately equidistant (the four O···O distances range from 3.37 to 3.48 Å). A similar orientation is observed on the left side of the X-ray structure (O···O = 2.98 and 3.47 Å), whereas the bottom right nitro group is twisted by approximately 90° from this arrangement.

The 20 smallest-volume *MOLPAK* packing arrangements [a total of 19^3 (6859) were created] were refined with the *WMIN* procedure (lattice energy minimization), which adjusts the search-probe orientation and position and unit-cell constants. The structure with the lowest lattice energy is generally the best (correct structure), but in a situation where experimental cell constants are available, these data can be used to indicate which of the hypothetical structures is most probably correct. Both the lattice energy and cell-parameter comparison criteria pointed to the same structure in the present case. Portions of the refined X-ray and predicted crystal structures are shown in Fig. 3. The predicted structure shown is based on the *AM*1-optimized search probe; differences in the C—NO_2 conformations alter the crystal packing somewhat and presumably are the cause of the orientation and position differences in the crystal structures. A *WMIN* refinement,

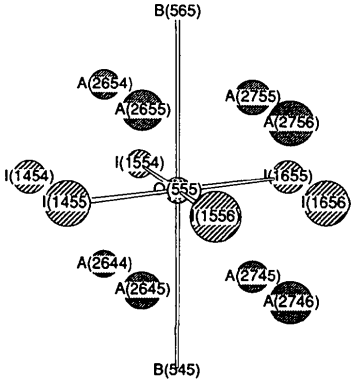
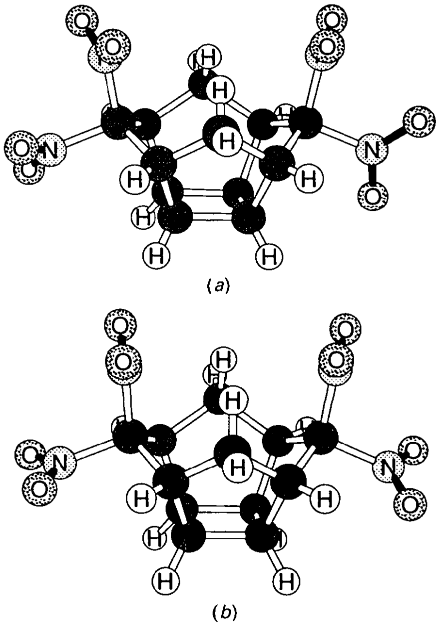


Fig. 1. Coordination geometry for space group $P2_1$. The three- and four-digit numbers are similar to *ORTEP* (Johnson, 1976) ADC codes. The central molecule is O(555) and the ± *b*-axis directions are indicated with B(565) and B(545). The first number of the four-digit codes refers to the symmetry operations: $1 = x,\ y,\ z$; $2 = -x,\ \frac{1}{2}+y,\ -z$. This coordination is characterized by six I (identity) molecules in the *ac* plane and four A (axis) molecules in planes above and below the *ac* plane. The positions labeled I(1655), I(1455), I(1556) and I(1554) show the *a* and *c* axes.



(*a*)

(*b*)

Fig. 2. *Chem*3D (Cambridge Scientific Computing, 1990) views of the refined X-ray (*a*) and *AM*1-optimized search probe (*b*) structures.

Table 2. *Comparison of the cell constants from the X-ray and predicted structures**

	X-ray	Δ_1 (%)	Δ_2 (%)
a (Å)	7.073	3.1	2.1
b (Å)	11.625	0.1	0.5
c (Å)	8.231	−0.3	−1.6
β (°)	96.79°	−4.7	−0.5

* X-ray = experimental cell constants. Δ_1 and Δ_2 give per cent differences between the predicted unit-cell parameters (1 and 2 indicate without and with *WMIN* C—NO$_2$ optimization). % = 100 × (predicted − X-ray)/X-ray.

which included optimization of the four C—NO$_2$ twist angles, yielded a hypothetical structure with an r.m.s. deviation of 0.26 Å from the refined X-ray structure [the deviation is from a least-squares fit of the 24 C, N and O atoms and 8 unit-cell corners (000, 100, 010 *etc.*) in the two structures]. The cell constant information for the experiment and two predicted structures is listed in Table 2. A structure-factor calculation with the predicted structure (*WMIN*-optimized C—NO$_2$ twist) and the 197 data to sin $\theta/\lambda = 0.324$ Å^{-1} ($\theta = 30°$) gave $R = 0.29$ and $wR = 0.37$ (no attempt was made to correct the model coordinates for differences between the experimental and predicted cell constants; this calculation was performed with the unmodified predicted fractional coordinates); several cycles of least-squares refinement with individual isotropic temperature factors and the 376 data to sin $\theta/\lambda = 0.417$ Å^{-1} ($\theta = 40°$) gave $R = 0.103$ and $wR = 0.122$. The refinement was completed in the usual way.

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
O1	−0.364 (1)	0.0620	0.670 (1)	5.3 (5)
O2	−0.538 (2)	0.206 (1)	0.591 (1)	7.6 (6)
O3	−0.306 (1)	−0.041 (1)	0.354 (1)	5.3 (5)
O4	−0.041 (1)	0.034 (1)	0.474 (1)	4.9 (5)
O5	0.049 (2)	0.452 (1)	−0.090 (1)	6.6 (6)
O6	−0.251 (1)	0.454 (1)	−0.154 (1)	7.0 (6)
O7	−0.060 (1)	0.177 (1)	−0.148 (1)	4.7 (4)
O8	0.167 (1)	0.220 (1)	0.043 (1)	5.2 (4)
N1	−0.415 (1)	0.140 (1)	0.574 (1)	3.8 (5)
N2	−0.206 (2)	0.037 (1)	0.416 (1)	3.8 (5)
N3	−0.113 (2)	0.418 (1)	−0.081 (1)	3.4 (5)
N4	0.003 (1)	0.227 (1)	−0.024 (1)	2.9 (4)
C1	−0.308 (1)	0.155 (1)	0.421 (1)	2.8 (4)
C2	−0.178 (1)	0.259 (1)	0.454 (1)	2.6 (4)
C3	−0.016 (1)	0.268 (1)	0.343 (1)	2.3 (4)
C4	−0.066 (1)	0.360 (1)	0.211 (1)	2.5 (5)
C5	−0.129 (1)	0.317 (1)	0.042 (1)	2.4 (4)
C6	−0.337 (1)	0.279 (1)	0.031 (1)	2.0 (4)
C7	−0.370 (1)	0.162 (1)	0.109 (1)	2.6 (5)
C8	−0.449 (1)	0.178 (1)	0.272 (1)	2.9 (5)
C9	−0.317 (2)	0.359 (1)	0.401 (1)	2.5 (4)
C10	−0.246 (1)	0.421 (1)	0.251 (1)	2.4 (4)
C11	−0.418 (1)	0.368 (1)	0.139 (1)	2.5 (4)
C12	−0.485 (1)	0.310 (1)	0.285 (1)	2.6 (4)

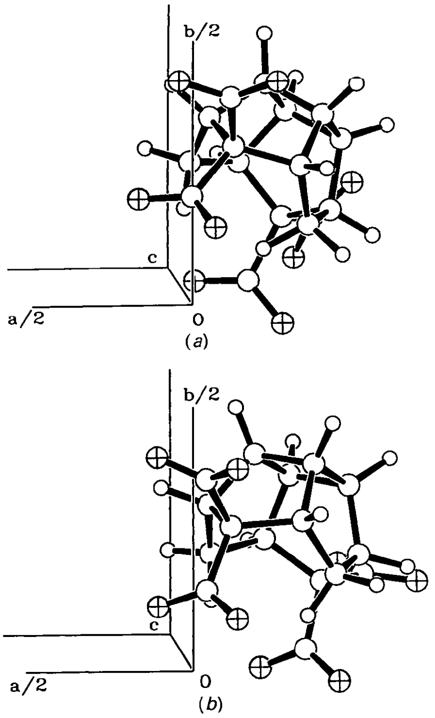
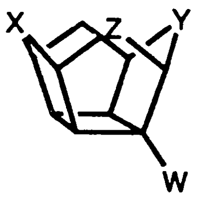


Fig. 3. *ORTEP* (Johnson, 1976) drawings of the X-ray (*a*) and predicted (*b*) crystal structures. The predicted structure shown was obtained from the *MOLPAK*/*WMIN* procedure and does not include refinement of the C—NO$_2$ conformations.

Discussion

An *ORTEP* (Johnson, 1976) drawing is shown in Fig. 4, fractional atomic coordinates are given in Table 3, and bond lengths, bond angles and selected torsion angles are given in Table 4. There is substantial similarity with the structures of other [4]peristylanes [(II)–(IX)], but the rather high parameter standard deviations in (I) preclude detailed comparisons. The structures of only two other [4]peristylanes [(VI) and (IX)] with sp^3 hybridization at all four crown positions (C1, C3, C5, C7) are known. The



(II): X = Y = C = O, Z = CH$_2$, W = H
(III): X = Y = C = O, Z = CH$_2$, W = NO$_2$
(IV): X = C = O, Y = C(NO$_2$)$_2$, Z = CH$_2$, W = H
(V): X = C = O, Y = C(NO$_2$)$_2$, Z = CH$_2$, W = NO$_2$
(VI): X = Y = C(NO$_2$)$_2$, Z = CH$_2$, W = NO$_2$
(VII): X = Y = Z = C = O, Z = CH$_2$, W = H
(VIII): X = Y = C = O, Z = C=C(CH$_3$)$_2$, W = H
(IX): X = Y = CHCH$_2$C(=O)C$_6$H$_5$, Z = CH$_2$, W = H

Table 4. *Geometric parameters* (Å, °)

O1—N1	1.24 (1)	C2—C3	1.55 (1)
O2—N1	1.18 (1)	C2—C9	1.56 (1)
O3—N2	1.23 (1)	C3—C4	1.53 (1)
O4—N2	1.21 (1)	C4—C5	1.50 (1)
O5—N3	1.21 (1)	C4—C10	1.52 (1)
O6—N3	1.16 (1)	C5—C6	1.52 (1)
O7—N4	1.22 (1)	C6—C7	1.53 (1)
O8—N4	1.22 (1)	C6—C11	1.52 (1)
N1—C1	1.56 (1)	C7—C8	1.52 (1)
N2—C1	1.55 (1)	C8—C12	1.56 (2)
N3—C5	1.57 (1)	C9—C10	1.56 (1)
N4—C5	1.54 (1)	C9—C12	1.54 (1)
C1—C2	1.52 (1)	C10—C11	1.56 (1)
C1—C8	1.51 (1)	C11—C12	1.50 (2)
O1—N1—O2	125 (1)	N3—C5—N4	100.8 (7)
O1—N1—C1	118 (1)	N3—C5—C4	107.9 (8)
O2—N1—C1	118 (1)	N3—C5—C6	108.9 (8)
O3—N2—O4	128 (1)	N4—C5—C4	115.1 (8)
O3—N2—C1	115 (1)	N4—C5—C6	114.2 (8)
O4—N2—C1	117 (1)	C4—C5—C6	109.4 (8)
O5—N3—O6	127 (1)	C5—C6—C7	115.0 (8)
O5—N3—C5	115 (1)	C5—C6—C11	101.4 (8)
O6—N3—C5	119 (1)	C7—C6—C11	105.6 (8)
O7—N4—O8	126 (1)	C6—C7—C8	110.7 (8)
O7—N4—C5	115.9 (9)	C1—C8—C7	114.7 (8)
O8—N4—C5	118.2 (9)	C1—C8—C12	102.3 (9)
N1—C1—N2	101.5 (8)	C7—C8—C12	105.1 (9)
N1—C1—C2	106.6 (9)	C2—C9—C10	108.3 (8)
N1—C1—C8	109.7 (8)	C2—C9—C12	107.8 (9)
N2—C1—C2	115.7 (8)	C10—C9—C12	88.6 (8)
N2—C1—C8	113.4 (9)	C4—C10—C9	108.2 (8)
C2—C1—C8	109.4 (9)	C4—C10—C11	107.8 (8)
C1—C2—C3	115.1 (9)	C9—C10—C11	89.4 (7)
C1—C2—C9	101.4 (8)	C6—C11—C10	107.1 (8)
C3—C2—C9	105.7 (9)	C6—C11—C12	110.0 (9)
C2—C3—C4	109.9 (8)	C10—C11—C12	90.2 (8)
C3—C4—C5	115.9 (9)	C8—C12—C9	106.8 (9)
C3—C4—C10	107.5 (7)	C8—C12—C11	108.3 (9)
C5—C4—C10	100.8 (8)	C9—C12—C11	91.9 (8)
O1—N1—C1—N2	17 (1)	O5—N3—C5—N4	−56 (1)
O1—N1—C1—C2	−105 (1)	O5—N3—C5—C4	65 (1)
O1—N1—C1—C8	137 (1)	O5—N3—C5—C6	−176 (1)
O2—N1—C1—N2	−164 (1)	O6—N3—C5—N4	124 (1)
O2—N1—C1—C2	74 (1)	O6—N3—C5—C4	−115 (1)
O2—N1—C1—C8	−44 (1)	O6—N3—C5—C6	3 (1)
O3—N2—C1—N1	81 (1)	O7—N4—C5—N3	−76 (1)
O3—N2—C1—C2	−164 (1)	O7—N4—C5—C4	168.4 (9)
O3—N2—C1—C8	−37 (1)	O7—N4—C5—C6	41 (1)
O4—N2—C1—N1	−99 (1)	O8—N4—C5—N3	97 (1)
O4—N2—C1—C2	16 (1)	O8—N4—C5—C4	−18 (1)
O4—N2—C1—C8	144 (1)	O8—N4—C5—C6	−146.0 (9)

X-ray derived crystal density of 1.682 g cm^{-3} is lower than the 1.70 g cm^{-3} reported from solvent density measurements (Waykole, Shen & Paquette, 1988). The relative densities of (I) and the 2,2,5,5,9-pentanitro derivative (VI; 1.731 g cm^{-3}; unpublished) are in line with the replacement of a basal H atom by NO_2. The crystal packing is normal. There are no intermolecular distances less than the appropriate van der Waals contact values.

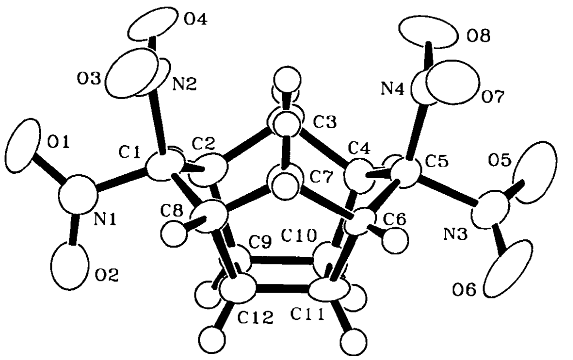


Fig. 4. *ORTEP* (Johnson, 1976) drawing. The C, N and O atoms are shown as 50% ellipsoids and the H atoms are drawn as spheres with $B = 1.5$ Å^2.

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