scheme is given in Fig. 1. The molecules are not linked together by hydrogen bonds. The shortest intermolecular distance N···C6^i is 3·320 (3) Å [(i) = x, $-y$, $z - 0{\cdot}5$]. The molecule is in the special position 4(c) of the space group $Pbcn$ possessing crystallographic symmetry C_2. The maximum deviation [0·071 (3) Å] of an atom from the least-squares plane through C1, C2, C3, N, C4, C5, C6, O is that of C2. The dihedral angle between this plane and the least-squares plane through the phenyl ring is 28·89 (5)°. The phenyl group geometries are normal.

There is an intramolecular N—H···O hydrogen bond with carbonyl oxygen [O···HN 1·85 (3), N···O 2·602 (3) Å]. The C=O bond distance of 1·256 (3) Å corresponds to the expected value according to the molecular formula (III) (Stephen & Trotter, 1988; Corkern, Fronczek, Gandour, Guo, Oliver & Watkins, 1988; Kaitner, Jovanovski & Janev, 1992). At the same time the N—C4 bond length of 1·347 (3) Å is very close to the value expected according to the formula (IV) (Wheatley, 1957; Morrow & Huddle, 1972). Some of the bond distances do not strictly belong to either formula (III) or (IV); C4—C5 1·370 (3), C5—C6 1·437 (3) and N—C3 1·383 (3) Å are midway between limiting values defining the above formulae.

This work was supported by the Federal (Belgrade) to BK and Republican (Zagreb, Croatia, and Skopje, Macedonia) Foundations for Scientific Research.

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Acta Cryst. (1992). C**48**, 131–133

Structure of 3,5-Di-*tert*-butyl-10-iodoprotoadamantane

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(*Received* 6 *February* 1991; *accepted* 27 *June* 1991)

Abstract. 6,7a-Di-*tert*-butyl-8-iodooctahydro-2,5-methano-1H-indene, $C_{18}H_{31}I$, $M_r = 374.35$, monoclinic, $P2_1$, $a = 9.095\,(1)$, $b = 7.087\,(1)$, $c = 14.149\,(2)$ Å, $\beta = 107.87\,(3)°$, $V = 867.9\,(2)$ Å^3, $Z = 2$, $D_x = 1.432$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 18.1$ cm^{-1}, $F(000) = 384$, $T = 293$ K, $R = 0.025$ for 1673 observed reflections [$F_o \geq 4\sigma(F_o)$]. The protoadamantane cage has a five-membered ring in the envelope conformation, one six-membered ring (with the I substituent) in a distorted chair conformation and the other six-membered ring (with two *tert*-butyl groups) in a boat conformation. The I atom is in an axial position. Molecules are joined by van der Waals contacts.

Introduction. Protoadamantanes have been accepted as intermediates in acid-catalyzed multiple rearrangements of diamondoid hydrocarbons (McKervey, 1980). The title compound was obtained by hydriodic-acid-catalyzed rearrangement of di-*tert*-butyladamantol (Duddeck, McKervey & Rosenbaum, 1990). The starting material and products were characterized by one- and two-dimensional NMR experiments including NOE difference spectra. The X-ray analysis of one of the products, reported in this paper, was undertaken in order to confirm the structure. The results obtained are in agreement with NMR data.

Experimental. Suitable crystals for X-ray structure analysis were grown from ethanol at 277 K over a

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few days. The X-ray data were collected using an Enraf–Nonius CAD-4F diffractometer in ω/θ scan mode with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å). The size of the crystal used was $0.35 \times 0.30 \times 0.30$ mm. The cell parameters were determined by least-squares refinement using setting angles for 22 reflections in the range $11 < \theta < 15°$. Intensities were collected for $2.5 < \theta < 27°$, the index range was $0 < h < 11$, $0 < k < 9$, $-18 < l < 18$; $\Delta\omega = (0.8 + 0.35\tan\theta)°$ and aperture setting 4 mm. Three intensity-controlled reflections were measured every hour. They revealed a loss of intensity of about 7.1%. A linear decay correction, and Lorentz and polarization corrections were carried out. An empirical absorption correction based on ψ scans of reflections $12\bar{2}$, $13\bar{2}$, $26\bar{4}$, $25\bar{5}$ and 043 was applied to the data. Relative transmission coefficients ranged from 0.950 to 0.999. 1837 reflections measured. Data reduction and refinement were by the Enraf–Nonius *SDP* package (B. A. Frenz & Associates, Inc., 1985). A fragment of the structure was obtained from the E map based on the phasing procedure in *MULTAN* (Main *et al.*, 1980). The complete structural model was obtained after a few subsequent difference Fourier syntheses. The refinement was by full-matrix least squares, minimizing $\sum w||F_o| - |F_c||$ with weights assigned according to Killean & Lawrence (1969). Scattering factors were those of Cromer & Waber (1974), and for H from Stewart, Davidson & Simpson (1965). Anomalous-scattering corrections were made according to Cromer (1974). H atoms were located from the difference Fourier map and refined with individual isotropic thermal parameters. However, the procedure was not successful for H51, H61 and the methyl groups of C13, C14 and C18. Their positions were calculated and refined with constraint of pivot-atom geometry. $wR = 0.028$, $S = 1.05$. Maximum shift/e.s.d. detected was 0.33 (for the y coordinate of C14). Residual density was $0.0 < \Delta\rho < 0.5$ e Å^{-3}. The absolute configuration was not determined. The R values calculated for both enantiomers are the same. The enantiomer was chosen in accordance with that described in Duddeck, McKervey & Rosenbaum (1990).

The molecular geometry was calculated by *PARST* (Nardelli, 1983). Calculations were carried out on a MicroVAX II in the X-ray laboratory, Ruder Bošković Institute, Zagreb. Final atomic coordinates and equivalent isotropic parameters of non-H atoms are listed in Table 1.*

Table 1. *Final atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}
I	0.89120 (1)	1.3452	0.68360 (3)	5.923 (7)
C1	1.0870 (5)	1.0502 (8)	0.6154 (3)	4.1 (1)
C2	1.1800 (4)	0.8643 (9)	0.6289 (3)	3.63 (8)
C3	1.3381 (4)	0.9080 (6)	0.7074 (3)	3.12 (8)
C4	1.3395 (4)	0.8259 (7)	0.8084 (2)	3.06 (7)
C5	1.1932 (4)	0.8729 (7)	0.8354 (2)	2.91 (7)
C6	1.1449 (5)	1.0798 (7)	0.8042 (3)	3.22 (8)
C7	1.2865 (5)	1.1953 (7)	0.8012 (3)	3.57 (9)
C8	1.3382 (5)	1.1301 (6)	0.7133 (3)	3.41 (9)
C9	1.2131 (6)	1.1918 (8)	0.6187 (3)	4.3 (1)
C10	1.0219 (5)	1.0812 (7)	0.7021 (3)	3.7 (1)
C11	1.4789 (4)	0.8306 (9)	0.6764 (3)	3.60 (8)
C12	1.4954 (5)	0.9262 (9)	0.5826 (3)	5.0 (1)
C13	1.6323 (4)	0.858 (1)	0.7593 (3)	4.51 (9)
C14	1.4576 (6)	0.6166 (8)	0.6535 (4)	5.1 (1)
C15	1.2017 (4)	0.8313 (9)	0.9448 (2)	3.20 (7)
C16	1.0371 (4)	0.839 (1)	0.9532 (3)	4.14 (8)
C17	1.3062 (6)	0.9634 (9)	1.0197 (3)	5.1 (1)
C18	1.2586 (5)	0.6271 (8)	0.9697 (3)	4.4 (1)

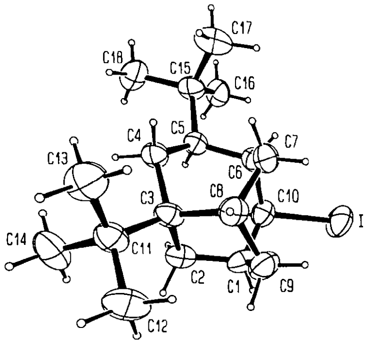

Fig. 1. An *ORTEP* (Johnson, 1976) plot of the molecule with the atom numbering.

Discussion. The molecule is shown in Fig. 1 and the molecular geometry is given in Table 2.

The molecule has sp^3 C atoms only; all C—C bond distances are in the range 1.512 (7)–1.555 (5) Å, except C3—C8 [1.577 (6) Å] and C3—C11 [1.574 (6) Å]. The thermal parameters do not suggest any higher thermal vibrations of these atoms.

In the five-membered ring, the mean bond angle is 102.9 (4)°, much lower than tetrahedral, while bond angles significantly larger than tetrahedral are associated with the *tert*-butyl groups. The C—I distance and the enlargement of the tetrahedral angles are similar to those reported for 2-iodoadamantane (Wahl, Greene & Bordner, 1974).

In this protoadamantane there is a five-membered ring in the envelope conformation (C1, C2, C3, C8, C9), one six-membered ring (C1, C9, C8, C7, C6, C10) in a distorted chair conformation, and another six-membered ring (C3, C4, C5, C6, C7, C8), with

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* Lists of H-atom coordinates, anisotropic thermal parameters, bond distances and angles, torsion angles, structure factors, and a packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54408 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0064]

Table 2. *Bond distances* (Å) *and selected angles* (°) *with e.s.d.'s in parentheses*

All other angles involving C and I atoms are in the range 108.0–110.0°.

C1—C2	1.545 (8)	C6—C10	1.532 (5)
C1—C9	1.513 (8)	C7—C8	1.531 (7)
C1—C10	1.533 (7)	C8—C9	1.531 (6)
C2—C3	1.555 (5)	C11—C12	1.538 (7)
C3—C4	1.539 (5)	C11—C13	1.535 (5)
C3—C8	1.577 (6)	C11—C14	1.550 (8)
C3—C11	1.574 (6)	C15—C16	1.540 (5)
C4—C5	1.529 (5)	C15—C17	1.512 (7)
C5—C6	1.555 (6)	C15—C18	1.541 (8)
C5—C15	1.555 (5)	I—C10	2.190 (5)
C6—C7	1.537 (7)		

C2—C1—C9	100.4 (4)	C2—C3—C11	112.6 (3)
C1—C2—C3	105.6 (4)	C8—C3—C11	112.0 (4)
C2—C3—C8	102.8 (3)	C3—C4—C5	112.9 (3)
C3—C8—C9	104.5 (3)	C4—C5—C15	115.2 (3)
C7—C8—C9	107.0 (4)	C6—C5—C15	112.6 (4)
C1—C9—C8	101.0 (4)	C1—C10—C6	113.8 (4)
C12—C11—C13	107.3 (4)	C3—C11—C12	113.0 (4)
C12—C11—C14	106.9 (4)	C3—C11—C13	111.7 (3)
C16—C15—C18	106.5 (5)	C5—C15—C17	114.2 (4)

two *tert*-butyl substituents, in the boat conformation. Both *tert*-butyl groups are in −antiperiplanar conformations [C5—C4—C3—C11 −171.6 (3), C3—C4—C5—C15 −167.7 (3)°]. The conformation for sequence C7—C6—C10—I is −synclinal [−80·6 (4)°].

Support of this research by the Council for Scientific Research of Croatia, and KFA Jülich, Germany, is gratefully acknowledged.

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Acta Cryst. (1992). **C48**, 133–135

Structure du Diammonium-1,3 Propane Bis(dihydrogénomonophosphate)

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(*Reçu le* 6 *mars* 1991, *accepté le* 3 *juillet* 1991)

Abstract. 1,3-Propanediammonium bis(dihydrogenmonophosphate), $C_3H_{12}N_2^{2+}.2H_2PO_4^-$, $M_r = 270.12$, monoclinic, Cc, $a = 18.543$ (3), $b = 4.561$ (4), $c = 15.342$ (3) Å, $\beta = 129.14$ (1)°, $V = 1006.5$ (7) Å^3, $Z = 4$, $D_m = 1.729$, $D_x = 1.782$ Mg m^{-3}, $\lambda(\text{Ag } K\alpha) = 0.56087$ Å, $\mu = 2.356$ mm^{-1}, $F(000) = 568$, $T = 298$ K, final $R = 0.027$ for 3362 independent reflexions. The structure shows a layer arrangement parallel to the b axis: planes of $[H_2PO_4]^-$ tetrahedra alternate with planes of $[(CH_2)_3(NH_3)_2]^{2+}$ groups.

0108-2701/92/010133-03$03.00