(Sheldrick, 1976). All calculations on laboratory micro-Vax I computer system. Atomic parameters given in Tables 1 and 2, selected bond distances and angles in Table 3,* the numbering scheme used is shown in Fig. 1.

Related literature. Hygric acid (I), a previously unknown naturally occurring compound in higher plants, has been found to accumulate in various species of the *Melaleuca* genus (Jones, Naidu, Paleg & Tiekink, 1988). (I) has been implicated in the biosynthesis of stachydrine (II), a compound which occurs widely in higher plants (Delaveau, Koudogbo & Pousset, 1972) and in algae (Blunden, Gorden, McLean & Guiry, 1982).

* Lists of structure factors, thermal parameters, H-atom parameters and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44999 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

BLUNDEN, G., GORDON, S. M., McLEAN, W. F. H. & GUIRY, M. D. (1982). *Bot. Marina*, **25**, 563–567.
BOER, J. L. DE & DUISENBERG, A. J. M. (1984). Enraf–Nonius CAD-4F diffractometer software update, February 1984. Groningen and Utrecht, The Netherlands.
CORNFORTH, J. W. & HENRY, A. J. (1952). *J. Chem. Soc.* pp. 601–603.
DELAVEAU, P., KOUDOGBO, B. & POUSSET, J.-L. (1972). *Phytochemistry*, **12**, 2893–2895.
JOHNSON, C. K. (1971). *ORTEP*II. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
JONES, G. P., NAIDU, B. P., PALEG, L. G. & TIEKINK, E. R. T. (1988). In preparation.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1986). *SHELXS*86. Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.

Acta Cryst. (1988). C44, 1671–1672

Structure of 1,4-Diiodocubane

By HERMAN L. AMMON

Department of Chemistry and Biochemistry and Center for Advanced Research in Biotechnology, University of Maryland, College Park, MD 20742, USA

CHANG S. CHOI

Energetics and Warheads Division, ARDEC, Picatinny Arsenal, NJ 07806, USA and Reactor Radiation Division, National Bureau of Standards, Gaithersburg, MD 20899, USA

AND SIVAKUMAR REDDY

Geocenters Inc., at Energetics and Warheads Division, ARDEC, Picatinny Arsenal, NJ 07806, USA

Abstract. 1,4-Diiodopentacyclo[4.2.0.0^{2,5}.0^{3,8}.0^{4,7}]-octane, $C_8H_6I_2$, $M_r = 355.9$, monoclinic, $P2_1/c$, $a = 7.137$ (1), $b = 7.269$ (2), $c = 8.991$ (2) Å, $\beta = 111.69$ (2)°, $V = 433.4$ (3) Å^3, $Z = 2$, $D_x = 2.73$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å (graphite monochromator), $\mu = 72.6$ cm^{-1}, $F(000) = 320$, $T = 293$ K, final $R = 0.036$ for 714 reflections with $I > 3\sigma(I)$. The molecule possesses a center of symmetry; the two I atoms are positioned on opposite corners of the cube.

Experimental. The compound (I) was synthesized by one of the authors (Reddy, 1988). Crystals obtained from methyl chloride and hexane solution; 0.27 × 0.20 × 0.23 mm rectangular shape used for X-ray measurements; Enraf–Nonius CAD-4 diffractometer;

Mo radiation with incident-beam monochromator; cell parameters from 25 reflections automatically centered in the range $4.7 < \theta < 25.7°$; $\theta–2\theta$ scan at variable θ speed of 1.03 to 8.24° min^{-1}; each scan recorded in 96 steps over the θ range of 1.5 × (1.2° + 0.35°tanθ) and subsequently processed with a modified Lehmann–Larsen profile analysis procedure (Lehmann & Larsen, 1974; Ammon, 1986); six standards measured at 200 data intervals; 923 data (includes standards and systematically absent reflections) measured from $\theta = 2$ to 25°; index range for $h, k, l = -8$ to 8, 0 to 8, -10 to 0; 830 unique reflections; 714 reflections with $I > 3\sigma(I)$; average change in standard intensities of 1.9% with a range of -1.4 to 4.3%; $R_{int} = 0.011$ (51 pairs). All crystallographic calculations performed with the

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

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

	x	y	z	B_{eq}
I	0.37474 (6)	−0.32515 (6)	0.14730 (6)	3.85 (3)
C1	0.1458 (9)	−0.1230 (9)	0.0574 (8)	2.9 (2)
C2	0.0613 (10)	−0.0003 (10)	0.1618 (8)	3.4 (2)
C3	0.0695 (10)	0.1664 (8)	0.0602 (10)	3.2 (2)
C4	0.1577 (10)	0.0484 (10)	−0.0416 (8)	3.4 (3)
H1	0.1075	−0.0059	0.2748	5 (2)
H2	0.1154	0.2845	0.1026	5 (2)
H3	0.2702	0.0788	−0.0713	9 (3)

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

The parameters involved with H atoms are given without e.s.d.'s. The atoms related by inversion symmetry are indicated by primes (').

|			
I—C1	2.123 (6)	C2—C3	1.531 (9)
C1—C3'	1.542 (9)	C2—C4'	1.577 (9)
C1—C4	1.551 (9)	C3—H2	0.95
C1—C2	1.567 (9)	C3—C4	1.548 (10)
C2—H1	0.95	C4—H3	0.96
C3'—C1—C4	91.3 (5)	H2—C3—C2	124.
C3'—C1—C2	91.2 (5)	H2—C3—C1'	125.
C3'—C1—I	123.5 (4)	H2—C3—C4	126.
C4—C1—C2	90.1 (5)	C2—C3—C1'	89.9 (5)
C4—C1—I	125.2 (4)	C2—C3—C4	91.6 (4)
C2—C1—I	125.3 (4)	C1'—C3—C4	90.1 (5)
H1—C2—C3	127.	H3—C4—C3	127.
H1—C2—C1	124.	H3—C4—C1	125.
H1—C2—C4'	126.	H3—C4—C2'	125.
C3—C2—C1	89.1 (5)	C3—C4—C1	89.2 (5)
C3—C2—C4'	90.8 (5)	C3—C4—C2'	90.7 (5)
C1—C2—C4'	88.1 (5)	C1—C4—C2'	88.0 (5)
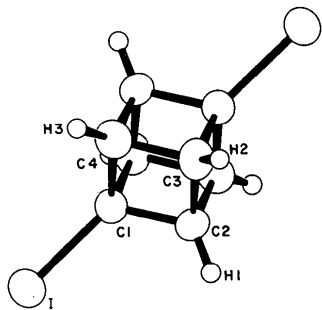
Fig. 1. *ORTEP* drawing (Johnson, 1965) of 1,4-diiodocubane molecule.

TEXSAN (1987) program system on DEC Microvax II computer; structure solved with Patterson methods; refinement with full-matrix least squares with anisotropic temperature factors for C, I, and isotropic terms for H (H's initially positioned from C-atom framework); minimization of $\Sigma w(F_o - F_c)^2$, $w = 1/\sigma^2(F_o)$, $\sigma^2(F_o) = (2\text{Lp}F_o)^{-2}[P + t^2(B_1 + B_2) + (0.03P)^2]$ where P = peak scan intensity, B_1 and B_2 are the two background intensities, t = scan time/total background time, Lp = Lorentz–polarization factor; secondary-extinc-

tion parameter [method of Zachariasen (1968)] refined, final extinction coefficient = 0.86 (6) × 10^{-5}; empirical absorption correction determined from systematic differences between F_o and F_c [method of Walker & Stuart (1983)], minimum and maximum corrections of 0.62 and 1.40; final S, R and wR values of 2.69, 0.036 and 0.053; maximum shift/e.s.d. of 0.88; minimum and maximum values in the final difference map of −1.07 and 0.79 e Å^{-3}. Atomic coordinates are listed in Table 1.* Bond lengths and angles are given in Table 2; an *ORTEP* (Johnson, 1965) drawing is shown in Fig. 1. The molecules are positioned on centers of symmetry.

(I)
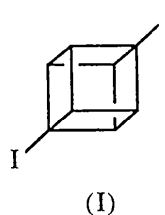
Related literature. There is considerable interest in the synthesis and chemistry of high-density energetic materials (Alster, Iyer & Marchand, 1983/84). Attention has been focused on the synthesis of highly nitrated cubane derivatives. (I) is an intermediate in the preparation of a dinitrocubane.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51004 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

ALSTER, J., IYER, S. & MARCHAND, A. P. (1983/84). *Working Group Meeting on Synthesis of High Density Energetic Materials*. Report. Armament Research and Development Center, Dover, NJ 07801, USA.
AMMON, H. L. (1986). *CAD4PROFILE*. Unpublished.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* A30, 580–584.
REDDY, S. (1988). In preparation.
TEXSAN (1987). *TEXRAY Structure Analysis System*, version 2.0. Molecular Structure Corp., College Station, TX 77804, USA.
WALKER, N. & STUART, D. (1983). *Acta Cryst.* A39, 158–166.
ZACHARIASEN, W. H. (1968). *Acta Cryst.* A24, 212–216.