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Dihydroxyisoasatone, $C_{24}H_{36}O_8$

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Abstract. Crystals grown from n-hexane–ethyl ether solution are monoclinic, $P2_1/c$, $a=10{\cdot}478$ (3), $b=17{\cdot}746$ (3), $c=14{\cdot}525$ (3) Å, $\beta=119{\cdot}76$ (4)°, $Z=4$, $D_{calc}=1{\cdot}282$, $D_{obs}=1{\cdot}279$ g cm^{-3}. The configuration of the compound has been established.

Introduction. The isolation of isoasatone and preliminary results have been reported in an earlier communication (Sasaki, Hirata, Yamamura, Chen, Hong & Hsu, 1973). A dihydroxyisoasatone was obtained as a reduced product of isoasatone, which was isolated from *Asarum taitonense* Hayata. Lattice constants and intensities were measured on a Hilger & Watts diffractometer (Ni-filtered Cu $K\alpha$ radiation, scintillation counter, θ–2θ scanning method). In all, 2440 independent non-zero reflexion data, which had F values greater than $3\sigma(F)$, were collected in the range, $\theta \leq 75°$. No absorption or extinction corrections were applied. A trial structure was obtained by direct methods with the computer program *MULTAN* (Germain, Main & Woolfson, 1971). The trial structure was refined by block-diagonal least-squares methods with anisotropic thermal parameters, and the final R value was 0·083. After the last cycle of the refinement most parameter shifts were less than $0{\cdot}1\sigma$. The final positional and thermal parameters are given in Table 1.*

All calculations were performed on a FACOM 230-60 at Nagoya University Computation Center. The atomic scattering factors used were from *International Tables for X-ray Crystallography* (1962).

* The table of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30402 (15 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Fractional atomic coordinates and thermal parameters for dihydroxyisoasatone*

Thermal parameters are of the form $T=\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{23}kl+\beta_{31}lh)]$. All values have been multiplied by 10^4.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{23}	β_{31}
C(1)	3491 (6)	874 (3)	2459 (4)	84 (7)	34 (2)	43 (3)	1 (6)	7 (5)	59 (8)
C(2)	2726 (6)	1082 (3)	1269 (4)	98 (7)	34 (2)	53 (4)	10 (7)	1 (5)	76 (9)
C(3)	2916 (6)	400 (4)	657 (4)	105 (7)	42 (2)	56 (4)	10 (7)	−6 (5)	84 (10)
C(4)	4386 (6)	783 (4)	867 (4)	100 (7)	38 (2)	47 (4)	6 (7)	2 (5)	67 (9)
C(5)	5168 (6)	285 (3)	1897 (4)	87 (7)	32 (2)	52 (3)	−2 (6)	−4 (5)	78 (8)
C(6)	3795 (6)	−246 (3)	1429 (4)	105 (7)	33 (2)	57 (4)	−3 (7)	−20 (5)	86 (9)
C(7)	3230 (7)	−539 (4)	2146 (5)	150 (9)	37 (2)	83 (5)	−4 (8)	−6 (6)	148 (12)
C(8)	2890 (6)	144 (4)	2656 (4)	88 (7)	38 (2)	53 (4)	1 (7)	0 (5)	74 (9)
C(9)	5157 (6)	728 (3)	2814 (4)	88 (7)	28 (2)	60 (4)	−8 (6)	−14 (5)	88 (9)
C(10)	5899 (6)	1502 (3)	2986 (5)	105 (7)	33 (2)	63 (4)	0 (7)	−11 (5)	94 (10)
C(11)	5034 (7)	2019 (4)	1983 (5)	140 (9)	32 (2)	73 (5)	8 (8)	9 (6)	113 (11)
C(12)	3865 (7)	1536 (4)	1088 (5)	126 (8)	38 (2)	55 (4)	27 (7)	22 (5)	97 (10)
C(13)	1149 (7)	1406 (4)	778 (5)	111 (8)	46 (3)	62 (4)	46 (8)	7 (6)	72 (10)
C(14)	1090 (7)	2132 (5)	1318 (6)	130 (9)	61 (3)	92 (6)	67 (10)	8 (8)	87 (13)
C(15)	386 (9)	2180 (6)	1866 (7)	170 (12)	95 (5)	99 (7)	65 (14)	−47 (10)	93 (16)
C(16)	6648 (6)	−56 (4)	2117 (5)	94 (7)	41 (2)	60 (4)	30 (7)	1 (5)	79 (9)
C(17)	7335 (8)	−590 (5)	3031 (6)	148 (10)	63 (4)	115 (7)	68 (10)	40 (8)	160 (14)
C(18)	8561 (10)	−506 (7)	3888 (7)	211 (14)	120 (7)	97 (7)	79 (16)	41 (11)	159 (17)
O(19)	4072 (4)	−854 (3)	901 (3)	111 (5)	41 (2)	84 (3)	−10 (5)	−46 (4)	98 (7)
C(20)	2780 (7)	−1238 (4)	101 (5)	132 (9)	42 (3)	79 (5)	−37 (8)	−52 (6)	79 (11)
O(21)	4265 (7)	−1034 (3)	2935 (5)	338 (12)	40 (2)	154 (5)	90 (8)	67 (5)	332 (15)
O(22)	3528 (4)	−1 (3)	3757 (3)	118 (5)	42 (2)	56 (3)	5 (5)	24 (3)	73 (6)
C(23)	3151 (8)	536 (5)	4341 (5)	223 (13)	61 (3)	62 (5)	18 (11)	−16 (7)	160 (14)
O(24)	1350 (4)	223 (3)	2201 (3)	91 (5)	51 (2)	63 (3)	0 (5)	8 (4)	82 (6)
C(25)	596 (7)	−403 (5)	2393 (6)	135 (9)	59 (3)	103 (6)	−52 (10)	19 (8)	142 (13)
O(26)	5925 (5)	1893 (3)	3854 (3)	151 (6)	34 (2)	74 (3)	−11 (5)	−25 (4)	119 (8)
C(27)	6792 (9)	1522 (5)	4891 (5)	221 (13)	52 (3)	48 (4)	3 (11)	−10 (6)	75 (13)
O(28)	7342 (4)	1380 (3)	3182 (3)	104 (5)	38 (2)	85 (3)	−21 (5)	−20 (4)	103 (7)
C(29)	8267 (8)	2055 (5)	3464 (7)	156 (11)	50 (3)	136 (8)	−88 (10)	−36 (8)	164 (16)
O(30)	4370 (6)	2648 (3)	2191 (4)	218 (8)	29 (2)	120 (4)	30 (6)	13 (4)	178 (11)
O(31)	3025 (5)	1963 (4)	135 (4)	161 (7)	55 (2)	75 (3)	54 (6)	57 (4)	123 (8)
C(32)	3819 (9)	2263 (5)	−349 (6)	230 (14)	77 (4)	114 (7)	45 (13)	101 (9)	232 (18)

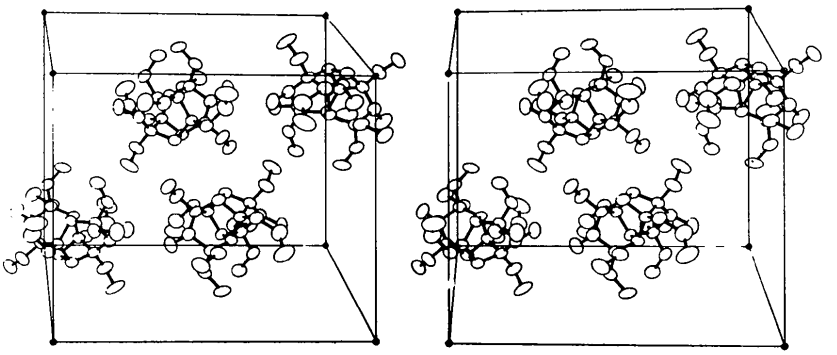

Fig. 1. Stereoscopic packing diagram. The directions of the axes are $y\rightarrow$, $z\uparrow$, and x out of the plane of the paper. One unit cell is outlined.

Discussion. A stereoscopic view of dihydroxyisoasatone molecules is shown in Fig. 1 (*ORTEP*, Johnson, 1965). The bond lengths are given in Fig. 2, and the bond angles in Table 2. The observed values for the bond lengths and angles are normal. The structural results are in agreement with those from spectroscopic measurements. The structure is cage-like and contains two four-membered rings. These rings are puckered and the dihedral angles between the planes C(3), C(2), C(12) and C(3), C(4), C(12), and between the planes C(3), C(4), C(5) and C(3), C(6), C(5) are 26·7° and 25·9°, respectively. The atoms C(1), C(2), C(3), C(6) all lie on the plane defined by the equation $0{\cdot}9078x+0{\cdot}3983y-0{\cdot}3368z=2{\cdot}7360$. There are no carbon–carbon intermolecular distances less than 3·5 Å. The closest intermolecular distances for carbon–oxygen and oxygen–oxygen are 3·31 and 2·79 Å, respectively.

Table 2. *Bond angles* (°)

The estimated standard deviations are about 0·5°.

C(2)—C(1)–C(8)	112·2	C(1)—C(8)—O(24)	108·0
C(2)—C(1)–C(9)	106·3	C(7)—C(8)—O(22)	107·7
C(8)—C(1)–C(9)	107·0	C(7)—C(8)—O(24)	109·5
C(1)—C(2)–C(3)	107·9	O(22)–C(8)—O(24)	110·0
C(1)—C(2)–C(12)	107·2	C(1)—C(9)—C(5)	106·4
C(1)—C(2)–C(13)	116·5	C(1)—C(9)—C(10)	107·2
C(3)—C(2)–C(12)	88·7	C(5)—C(9)—C(10)	111·9
C(3)—C(2)–C(13)	115·5	C(9)—C(10)–C(11)	111·0
C(12)–C(2)–C(13)	117·3	C(9)—C(10)–O(26)	111·4
C(2)—C(3)–C(4)	86·8	C(9)—C(10)–O(28)	107·6
C(2)—C(3)–C(6)	111·0	C(11)–C(10)–O(26)	106·5
C(4)—C(3)–C(6)	90·4	C(11)–C(10)–O(28)	110·1
C(3)—C(4)–C(5)	86·5	O(26)–C(10)–O(28)	110·4
C(3)—C(4)–C(12)	90·1	C(10)–C(11)–C(12)	108·1
C(5)—C(4)–C(12)	111·2	C(10)–C(11)–O(30)	112·2
C(4)—C(5)–C(6)	89·6	C(12)–C(11)–O(30)	110·4
C(4)—C(5)–C(9)	108·0	C(2)—C(12)–C(4)	88·1
C(4)—C(5)–C(16)	114·4	C(2)—C(12)–C(11)	120·1
C(6)—C(5)–C(9)	106·4	C(2)—C(12)–O(31)	106·3
C(6)—C(5)–C(16)	117·7	C(4)—C(2)—C(11)	116·5
C(9)—C(5)–C(16)	117·2	C(4)—C(12)–O(31)	111·7
C(3)—C(6)–C(5)	87·5	C(11)–C(12)–O(31)	111·9
C(3)—C(6)–C(7)	117·0	C(2)—C(13)–C(14)	112·5
C(3)—C(6)–O(19)	112·1	C(13)–C(14)–C(15)	122·6
C(5)—C(6)–C(7)	119·6	C(5)—C(16)–C(17)	115·1
C(5)—C(6)–O(19)	107·5	C(16)–C(17)–C(18)	126·9
C(7)—C(6)–O(19)	111·1	C(6)—C(19)–C(20)	115·3
C(6)—C(7)–C(8)	108·7	C(8)—O(22)–C(23)	115·1
C(6)—C(7)–O(21)	111·3	C(8)—O(24)–C(25)	115·6
C(8)—C(7)–O(21)	111·2	C(10)–O(26)–C(27)	114·7
C(1)—C(8)–C(7)	111·1	C(10)–O(28)–C(29)	115·2
C(1)—C(8)–O(22)	111·0	C(12)–O(31)–C(32)	116·3

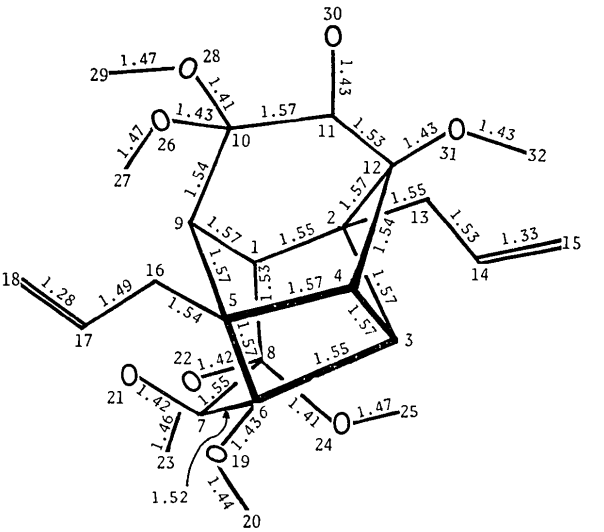

Fig. 2. Bond lengths. The estimated standard deviations are about 0·008 Å.

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