463.   *The Structure of Cedrelone: X-Ray Analysis of Cedrelone Iodoacetate.*[1]

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In order to determine the molecular structure of cedrelone, the principal crystalline constituent of the heartwood of *Cedrela toona*, we have carried out a crystal-structure analysis of the heavy-atom derivative, cedrelone iodoacetate.   The crystals are orthohombic, space group $P2_12_12_1$, with four molecules of $C_{28}H_{31}IO_6$ in the unit cell of dimensions $a = 6\cdot97$, $b = 27\cdot44$, $c = 13\cdot74$ Å.   Phase determination was based initially on the iodine atom, and Fourier and least-squares refinement methods were employed in the determination of the atomic co-ordinates.   Our final results define the constitution and relative stereochemistry of cedrelone iodoacetate to be as in (I); cedrelone is therefore assigned formula (II).

CEDRELONE, the principal crystalline constituent of the heartwood of the Indian tree *Cedrela toona*, was first isolated by Parihar and Dutt,[2] who favoured the molecular formula $C_{25}H_{30}O_5$ and reported the presence of a lactone ring, a phenolic hydroxyl and a ketone group, and an ethylenic double bond.   Later chemical and spectroscopic work at Glasgow,[1] Madras,[3] and Zurich [3] showed that the correct molecular formula is $C_{26}H_{30}O_5$ and that the molecule contains an αβ-unsaturated ketone, a β-monosubstituted furan ring, and an

[1] For a preliminary report see Grant, Hamilton, Hamor, Hodges, McGeachin, Raphael, Robertson, and Sim, *Proc. Chem. Soc.*, 1961, 444.
[2] Parihar and Dutt, *J. Indian Chem. Soc.*, 1950, **27**, 77.
[3] Gopinath, Govindachari, Parthasarathy, Viswanathan, Arigoni, and Wildman, *Proc. Chem. Soc.*, 1961, 446.

enolised α-diketone grouping similar in environment to that of diosphenols in the limonin series.[4]

No further chemical information about cedrelone was available to us when we began an X-ray analysis of cedrelone iodoacetate,[5] and a direct determination of the crystal structure of this derivative was effected by means of the usual phase-determining heavy-atom method.[6] A number of three-dimensional electron-density distributions were evaluated, and this was followed by least-squares refinement of the positional and thermal atomic parameters. At the end the value of R, the average discrepancy between measured and calculated structure amplitudes, was 17·5% over 1163 reflexions.

Our final results establish the constitution and stereochemistry (apart from absolute configuration) of cedrelone iodoacetate to be as in (I); it follows that cedrelone is represented by formula (II). The independent chemical studies at Glasgow,[1,5] Madras,[3] and Zurich[3] are consistent with this structure. Cedrelone, like limonin[4,7] (III) and
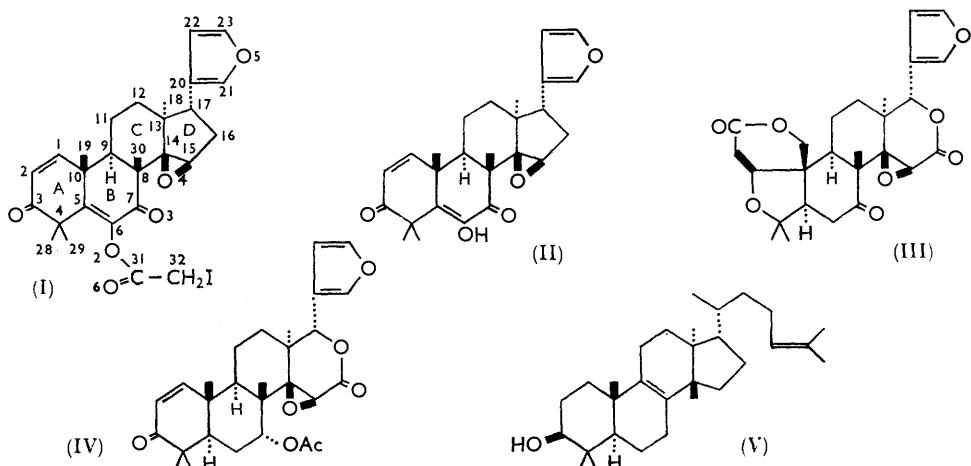
gedunin[8,9] (IV), is clearly a triterpenoid of the euphol[10] (V) type and its biogenesis presumably follows the route proposed for limonin.[4]

The final electron-density distribution for cedrelone iodoacetate is shown in Fig. 1 as superimposed contour sections drawn parallel to (001) and covering the region of one molecule; the corresponding atomic arrangement is illustrated in Fig. 2. The inter-atomic distances and valency angles calculated from the final atomic co-ordinates (see Table 1) are listed in Table 2. The standard deviations of the final atomic co-ordinates were estimated in the usual manner from the least-squares residuals (see Experimental section) and are shown in Table 3; from the results the average estimated standard deviation (e.s.d.) of a carbon–carbon bond is about 0·09 Å and of a carbon–oxygen bond about 0·07 Å. The average e.s.d. of a valency angle is about 4°.

The average carbon–carbon single-bond length is 1·55 Å, in good agreement with the value of 1·545 Å in diamond. The average carbon–carbon double-bond length, 1·35 Å,

[4] Barton, Pradhan, Sternhell, and Templeton, *J.*, 1961, 255; Arigoni, Barton, Corey, and Jeger, in collaboration with Cagliotti, Dev, Ferini, Glazier, Melera, Pradhan, Schaffner, Sternhell, Templeton, and Tobinaga, *Experientia*, 1960, **16**, 41.

[5] Hodges, McGeachin, and Raphael, following paper.

[6] Robertson and Woodward, *J.*, 1937, 219; 1940, 36; Sim, in " Computing Methods and the Phase Problem in X-ray Crystal Analysis," ed. Pepinsky, Robertson, and Speakman, Pergamon Press, Oxford, 1961, p. 227.

[7] Arnott, Davie, Robertson, Sim, and Watson, *Experientia*, 1960, **16**, 49; *J.*, 1961, 4183.

[8] Akisanya, Bevan, Hirst, Halsall, and Taylor, *J.*, 1960, 3827.

[9] Sutherland, Sim, and Robertson, *Proc. Chem. Soc.*, 1962, 222.

[10] Barton, McGhie, Pradhan, and Knight, *Chem. and Ind.*, 1954, 1325; *J.*, 1955, 876.
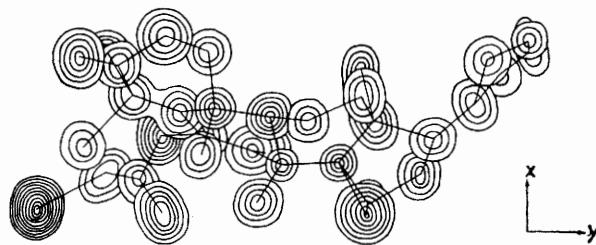
FIG. 1. The final three-dimensional electron-density distribution for cedrelone iodo-
acetate shown by means of superimposed contour sections drawn parallel to (001)
and covering the region of one molecule. Contour interval 1 eÅ^{-3}, starting from
the 2 eÅ^{-3} level, except around the iodine atom where the interval is 5 eÅ^{-3}.
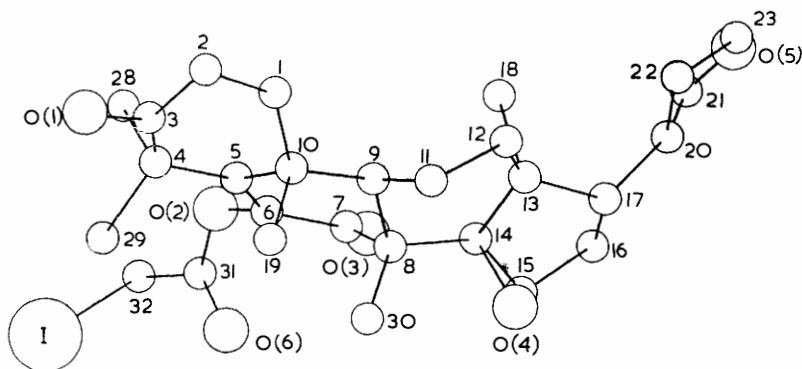
FIG. 2. The arrangement of atoms corresponding to Fig. 1.

TABLE 1.

Atomic co-ordinates and isotropic temperature factors.

(Origin of co-ordinates as in " International Tables." *)

Atom	x/a	y/b	z/c	B	Atom	x/a	y/b	z/c	B
C(1)	−0·3157	−0·0520	0·1550	4·5	C(19) ...	0·0544	−0·0578	0·1154	4·3
C(2)	−0·3783	−0·0957	0·1406	4·9	C(20) ...	−0·1626	0·2006	0·3251	5·7
C(3)	−0·2634	−0·1337	0·1687	6·6	C(21) ...	−0·2756	0·2147	0·4005	8·3
C(4)	−0·1512	−0·1311	0·2654	4·1	C(22) ...	−0·3139	0·2093	0·2532	6·4
C(5)	−0·0985	−0·0781	0·2787	3·9	C(23) ...	−0·4079	0·2487	0·2789	8·9
C(6)	−0·0131	−0·0596	0·3679	4·3	C(28) ...	−0·2911	−0·1504	0·3437	6·1
C(7)	0·0318	−0·0086	0·3925	5·8	C(29) ...	0·0410	−0·1661	0·2702	7·5
C(8)	0·0840	0·0183	0·2986	3·4	C(30) ...	0·2710	0·0030	0·2637	4·5
C(9)	−0·0851	0·0093	0·2311	3·1	C(31) ...	0·1412	−0·1035	0·4855	5·7
C(10) ...	−0·1186	−0·0424	0·1949	6·0	C(32) ...	0·1435	−0·1425	0·5768	5·6
C(11) ...	−0·0764	0·0460	0·1412	6·7	O(1) ...	−0·2808	−0·1767	0·1316	7·8
C(12) ...	−0·1651	0·0958	0·1854	5·7	O(2)......	−0·0223	−0·0932	0·4444	5·0
C(13) ...	−0·0779	0·1067	0·2925	4·5	O(3)......	0·0597	0·0047	0·4678	5·0
C(14) ...	0·0798	0·0744	0·3175	3·8	O(4)......	0·2559	0·0974	0·2779	4·0
C(15) ...	0·0235	0·1024	0·3824	4·8	O(5)......	−0·3849	0·2467	0·3740	8·3
C(16) ...	0·1063	0·1495	0·3960	5·5	O(6)......	0·2847	−0·0888	0·4452	5·9
C(17) ...	−0·0109	0·1585	0·3085	4·6	I	0·2808	−0·2054	0·5353	6·0
C(18) ...	−0·2785	0·0931	0·3694	3·4					

* " International Tables for *X*-Ray Crystallography," The Kynoch Press, Birmingham, 1952,
Vol. I.

TABLE 2.

Interatomic distances (Å) and angles.

Intramolecular bonded distances

C(1)–C(2)	1·29	C(6)–C(7)	1·48	C(11)–C(12)	1·62	C(17)–C(20)	1·58
C(1)–C(10)	1·50	C(6)–O(2)	1·40	C(12)–C(13)	1·62	C(20)–C(21)	1·36
C(2)–C(3)	1·57	C(7)–C(8)	1·53	C(13)–C(14)	1·45	C(20)–C(22)	1·47
C(3)–C(4)	1·54	C(7)–O(3)	1·11	C(13)–C(17)	1·51	C(21)–O(5)	1·22
C(3)–O(1)	1·29	C(8)–C(9)	1·52	C(13)–C(18)	1·79	C(22)–C(23)	1·31
C(4)–C(5)	1·51	C(8)–C(14)	1·56	C(14)–C(15)	1·55	C(23)–O(5)	1·32
C(4)–C(28)	1·55	C(8)–C(30)	1·45	C(14)–O(4)	1·48	C(31)–C(32)	1·65
C(4)–C(29)	1·65	C(9)–C(10)	1·52	C(15)–C(16)	1·54	C(31)–O(2)	1·30
C(5)–C(6)	1·45	C(9)–C(11)	1·59	C(15)–O(4)	1·46	C(31)–O(6)	1·21
C(5)–C(10)	1·52	C(10)–C(19)	1·68	C(16)–C(17)	1·47	C(32)–I	2·05

Intramolecular non-bonded distances

C(1) · · · C(4)	2·89	C(6) · · · C(29)	3·24	C(10) · · · O(1)	3·95	C(16) · · · C(22)	3·89
C(1) · · · C(11)	3·17	C(6) · · · C(30)	2·99	C(10) · · · O(2)	3·76	C(17) · · · C(23)	3·74
C(1) · · · C(28)	3·75	C(6) · · · C(32)	3·82	C(10) · · · O(3)	4·15	C(17) · · · O(4)	2·54
C(1) · · · O(1)	3·44	C(6) · · · O(6)	2·47	C(11) · · · C(17)	3·87	C(17) · · · O(5)	3·67
C(2) · · · C(5)	2·76	C(7) · · · C(10)	3·06	C(11) · · · C(18)	3·67	C(18) · · · C(20)	3·12
C(2) · · · C(9)	3·74	C(7) · · · C(11)	3·84	C(11) · · · C(19)	3·01	C(18) · · · C(21)	3·37
C(2) · · · C(19)	3·21	C(7) · · · C(13)	3·53	C(11) · · · C(30)	3·18	C(18) · · · C(22)	3·58
C(2) · · · C(28)	3·23	C(7) · · · C(15)	3·33	C(11) · · · O(4)	3·30	C(18) · · · O(3)	3·64
C(2) · · · C(29)	3·93	C(7) · · · C(18)	3·54	C(12) · · · C(15)	3·83	C(18) · · · O(4)	3·93
C(3) · · · C(6)	3·83	C(7) · · · C(31)	3·00	C(12) · · · C(16)	3·76	C(19) · · · C(29)	3·65
C(3) · · · C(10)	2·72	C(7) · · · O(4)	3·66	C(12) · · · C(20)	3·45	C(19) · · · C(30)	3·04
C(3) · · · C(19)	3·13	C(7) · · · O(6)	2·91	C(12) · · · C(22)	3·41	C(28) · · · C(31)	3·81
C(4) · · · C(9)	3·91	C(8) · · · C(12)	3·15	C(12) · · · O(4)	3·20	C(28) · · · O(1)	3·00
C(4) · · · C(19)	3·22	C(8) · · · C(16)	3·84	C(13) · · · C(21)	3·59	C(28) · · · O(2)	2·81
C(4) · · · C(31)	3·72	C(8) · · · C(17)	3·90	C(13) · · · C(22)	3·31	C(29) · · · C(31)	3·49
C(4) · · · O(2)	2·82	C(8) · · · C(18)	3·39	C(13) · · · C(30)	3·76	C(29) · · · O(1)	2·96
C(5) · · · C(8)	2·95	C(8) · · · O(2)	3·74	C(13) · · · O(3)	3·82	C(29) · · · O(2)	3·15
C(5) · · · C(11)	3·90	C(9) · · · C(15)	3·93	C(14) · · · C(20)	3·85	C(29) · · · O(6)	3·63
C(5) · · · C(30)	3·41	C(9) · · · C(18)	3·27	C(14) · · · O(3)	2·82	C(30) · · · O(3)	3·17
C(5) · · · C(31)	3·37	C(9) · · · O(3)	3·41	C(15) · · · C(18)	3·51	C(30) · · · O(4)	2·60
C(5) · · · O(1)	3·61	C(9) · · · O(4)	3·45	C(15) · · · C(20)	3·89	C(30) · · · O(6)	3·55
C(5) · · · O(3)	3·62	C(10) · · · C(12)	3·81	C(15) · · · C(30)	3·20	C(31) · · · O(3)	3·03
C(5) · · · O(6)	3·53	C(10) · · · C(14)	3·88	C(15) · · · O(3)	3·14	O(2) · · · O(3)	2·76
C(6) · · · C(9)	2·71	C(10) · · · C(28)	3·79	C(16) · · · C(18)	3·12	O(3) · · · O(4)	3·89
C(6) · · · C(19)	3·50	C(10) · · · C(29)	3·72	C(16) · · · C(21)	3·21	O(3) · · · O(6)	3·02
C(6) · · · C(28)	3·17	C(10) · · · C(30)	3·13				

Intermolecular distances (<4 Å)

C(32) · · · O(4)$_\mathrm{I}$	3·11	C(23) · · · C(29)$_\mathrm{IV}$	3·53	C(32) · · · C(22)$_\mathrm{III}$	3·81	O(5) · · · C(29)$_\mathrm{IV}$	3·92
O(5) · · · O(1)$_\mathrm{II}$	3·14	O(6) · · · C(11)$_\mathrm{I}$	3·57	C(23) · · · C(28)$_\mathrm{II}$	3·86	C(22) · · · C(29)$_\mathrm{IV}$	3·93
C(23) · · · O(1)$_\mathrm{II}$	3·23	O(3) · · · C(19)$_\mathrm{I}$	3·67	C(32) · · · C(12)$_\mathrm{III}$	3·87	O(2) · · · C(12)$_\mathrm{III}$	3·96
O(3) · · · C(1)$_\mathrm{III}$	3·35	O(3) · · · C(2)$_\mathrm{III}$	3·67	C(18) · · · C(2)$_\mathrm{III}$	3·88	C(20) · · · C(29)$_\mathrm{IV}$	3·98
C(21) · · · O(1)$_\mathrm{III}$	3·36	C(15) · · · C(19)$_\mathrm{I}$	3·76				

The subscripts refer to the following positions:

I $\frac{1}{2} - x,\ -y,\ \frac{1}{2} + z$. II $-1 - x,\ \frac{1}{2} + y,\ \frac{1}{2} - z$. III $-\frac{1}{2} - x,\ -y,\ \frac{1}{2} + z$. IV $-x,\ \frac{1}{2} + y,\ \frac{1}{2} - z$.

Valency angles

C(2)C(1)C(10)	122°	C(6)C(7)O(3)	124°	C(9)C(11)C(12)	103°	C(15)C(16)C(17)	110°
C(1)C(2)C(3)	118	C(8)C(7)O(3)	126	C(11)C(12)C(13)	111	C(13)C(17)C(16)	98
C(2)C(3)C(4)	120	C(7)C(8)C(9)	104	C(12)C(13)C(14)	113	C(13)C(17)C(20)	120
C(2)C(3)O(1)	122	C(7)C(8)C(14)	110	C(12)C(13)C(17)	115	C(16)C(17)C(20)	112
C(4)C(3)O(1)	115	C(7)C(8)C(30)	110	C(12)C(13)C(18)	102	C(17)C(20)C(21)	135
C(3)C(4)C(5)	106	C(9)C(8)C(14)	105	C(14)C(13)C(17)	108	C(17)C(20)C(22)	120
C(3)C(4)C(28)	105	C(9)C(8)C(30)	117	C(14)C(13)C(18)	109	C(21)C(20)C(22)	93
C(3)C(4)C(29)	115	C(14)C(8)C(30)	111	C(17)C(13)C(18)	111	C(20)C(21)O(5)	110
C(5)C(4)C(28)	114	C(8)C(9)C(10)	118	C(8)C(14)C(13)	125	C(20)C(22)C(23)	108
C(5)C(4)C(29)	111	C(8)C(9)C(11)	110	C(8)C(14)C(15)	125	C(22)C(23)O(5)	100
C(28)C(4)C(29)	107	C(10)C(9)C(11)	110	C(8)C(14)O(4)	110	C(32)C(31)O(2)	119
C(4)C(5)C(6)	123	C(1)C(10)C(5)	104	C(13)C(14)C(15)	109	C(32)C(31)O(6)	124
C(4)C(5)C(10)	120	C(1)C(10)C(9)	115	C(13)C(14)O(4)	106	O(2)C(31)O(6)	117
C(6)C(5)C(10)	117	C(1)C(10)C(19)	112	C(15)C(14)O(4)	58	C(31)C(32)I	110
C(5)C(6)C(7)	128	C(5)C(10)C(9)	110	C(14)C(15)C(16)	98	C(6)O(2)C(31)	115
C(5)C(6)O(2)	113	C(5)C(10)C(19)	105	C(14)C(15)O(4)	59	C(14)O(4)C(15)	63
C(7)C(6)O(2)	118	C(9)C(10)C(19)	110	C(16)C(15)O(4)	106	C(21)O(5)C(23)	114
C(6)C(7)C(8)	109						

TABLE 3.

Standard deviations of the final atomic co-ordinates (Å).

Atom	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	Atom	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	Atom	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
C(1)	0·059	0·042	0·046	C(13)	0·054	0·047	0·044	C(29)	0·075	0·050	0·055
C(2)	0·058	0·045	0·044	C(14)	0·053	0·047	0·041	C(30)	0·063	0·045	0·044
C(3)	0·069	0·042	0·043	C(15)	0·069	0·038	0·043	C(31)	0·052	0·045	0·056
C(4)	0·051	0·043	0·047	C(16)	0·059	0·040	0·050	C(32)	0·057	0·058	0·051
C(5)	0·049	0·038	0·044	C(17)	0·055	0·041	0·049	O(1)	0·051	0·031	0·032
C(6)	0·061	0·040	0·044	C(18)	0·051	0·036	0·047	O(2)	0·037	0·028	0·030
C(7)	0·064	0·051	0·049	C(19)	0·053	0·048	0·050	O(3)	0·037	0·028	0·033
C(8)	0·056	0·041	0·046	C(20)	0·059	0·048	0·043	O(4)	0·033	0·024	0·027
C(9)	0·047	0·035	0·048	C(21)	0·069	0·058	0·055	O(5)	0·078	0·042	0·058
C(10)	0·072	0·050	0·048	C(22)	0·050	0·046	0·053	O(6)	0·042	0·026	0·028
C(11)	0·068	0·049	0·055	C(23)	0·084	0·046	0·063	I	0·004	0·003	0·004
C(12)	0·063	0·042	0·051	C(28)	0·067	0·046	0·051				
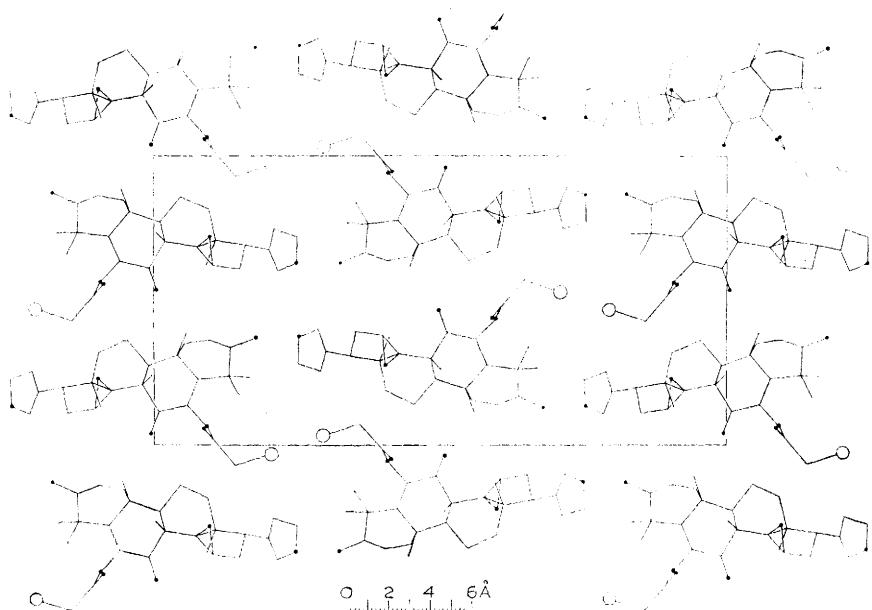
FIG. 3.   The crystal structure of cedrelone iodoacetate as viewed in projection along the *a*-axis.
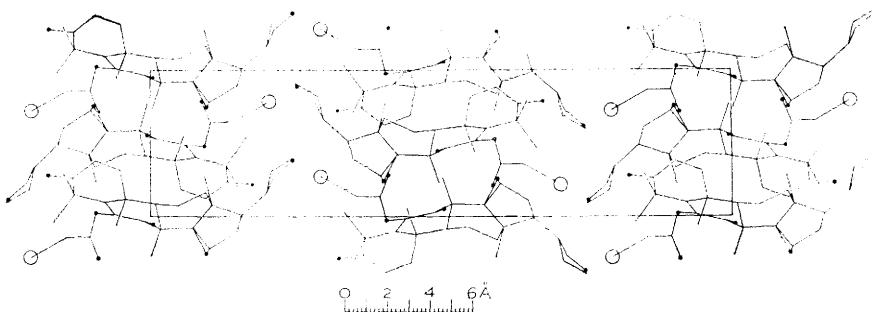
FIG. 4.   The crystal structure of cedrelone iodoacetate as viewed in projection along the *c*-axis.

does not differ significantly from the value of 1·334 Å in ethylene.[11] The average carbon–oxygen double-bond distance in the carbonyl groups of 1·20 Å compares favourably with the values of 1·212 Å in parabanic acid,[12] 1·222 Å in *p*-benzoquinone,[13] and 1·195 Å in tetrachloro-*p*-benzoquinone.[14] In the epoxide ring the average carbon–oxygen bond length is 1·47 Å, while the values quoted for ethylene oxide,[15] cyclopentene oxide,[16] and clerodin bromo-lactone [17] are 1·44, 1·47, and 1·49 Å, respectively. The bond lengths in the iodoacetate group show no significant deviation from the expected values.

The strong steric interaction between axial 1,3-methyl groups is reflected in the non-bonded distance of 3·04 Å between C(19) and C(30); on the basis of standard bond lengths and valency angles the distance between these atoms is expected to be only 2·54 Å. The steric interaction between the 28- and 29-methyl groups and the oxygen substituent at position 6 is presumably the reason why ring A adopts a half-boat conformation. Ring C is locked in a boat conformation by the β-oriented epoxide group.

The arrangement of the molecules in the crystal as viewed in projection along the *a*-axis is shown in Fig. 3; the projection along the *c* axis is shown in Fig. 4. The inter-molecular contacts (see Table 2) are all greater than 3 Å and correspond to normal van der Waals interactions.

EXPERIMENTAL

Crystal Data.—Cedrelone iodoacetate, $C_{28}H_{31}O_6I$; $M = 590·4$; m. p. 149—150°. Ortho-rhombic, $a = 6·97$, $b = 27·44$, $c = 13·74$ Å, $U = 2628$ Å³, $D_m = 1·498$ g. cm.⁻³ (by flotation), $Z = 4$, $D_c = 1·490$ g.cm.⁻³. Space group $P2_12_12_1(D_2^4)$. Absorption coefficient for *X*-rays ($\lambda = 1·542$ Å) $\mu = 108$ cm.⁻¹. Total number of electrons in the unit cell $= F(000) = 1200$.

The crystals were grown from solution in aqueous ethanol and were obtained in the form of thin plates with (010) strongly developed.

Experimental Measurements.—Rotation, oscillation, and moving-film photographs were taken with copper-K_α ($\lambda = 1·542$ Å) radiation. The cell dimensions were obtained from rotation and equatorial layer line Weissenberg photographs. The space group was determined uniquely from the systematic halvings in the *X*-ray spectra. For the intensity measurements small crystals were used, completely bathed in a uniform beam of *X*-rays. No corrections for absorption were applied. Photographic records of the 0*kl*–5*kl* and *hk*0–*hk*6 spectra were obtained by means of an equi-inclination Weissenberg camera. For correlation of strong and weak reflections the multiple-film technique [18] was used. The intensities were estimated visually and were corrected by the usual factors (Lorentz, polarisation, and rotation [19]). The various layers were placed on the same relative scale by comparison of common reflections on different photographs; the absolute scale was obtained at a later stage by correlation with the calculated structure amplitudes, $|F_c|$. In all only 1163 independent structure amplitudes were measured (Table 4), the low total being a consequence of the non-appearance of reflections beyond moderate values of sin θ. This effect (*i.e.*, a high temperature factor, *B*) probably indicates some disorder in the molecular packing in the crystal. In addition to the 1163 observed reflections 122 unobserved reflections at low values of sin θ were included in the structure analysis, each being assigned a value of $|F_o|$ equal to half the minimum $|F|$ locally observable. A total of 1285 reflections was therefore employed in the determination of the structure.

The crystal density was measured by flotation of the crystals in carbon tetrachloride–light petroleum.

Structure Analysis.—The co-ordinates of the iodine atom were determined initially from two-dimensional Patterson syntheses along the *a*- and the *c*-crystal axes. These projections are shown in Figs. 5 and 6.

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TABLE 4.

Measured and calculated values of the structure factors.

Column group 1

h	k	l	F_o	F_c	$\alpha°$
0	0	2	43	20	180
		4	48	57	0
		6	24	23	0
		10	42	45	180
0	1	1	94	157	90
		2	52	61	270
		3	242	278	90
		4	10	4	90
		5	72	73	90
		6	53	49	270
		7	10	22	270
		8	51	51	90
		9	51	54	270
		10	26	15	270
		11	58	51	270
0	2	1	43	22	180
		2	162	143	180
		3	26	29	180
		4	64	72	180
		5	55	57	180
		7	47	47	180
		8	20	11	0
		10	38	32	0
		11	35	41	180
0	3	1	69	59	270
		2	15	28	270
		3	21	18	270
		4	142	142	270
		5	62	56	270
		6	98	85	270
		7	21	19	270
		8	92	90	270
		10	48	44	270
0	4	0	18	8	180
		1	55	60	0
		2	72	71	0
		3	166	173	0
		4	80	71	0
		5	137	148	0
		7	110	96	0
		9	60	62	0
		11	20	25	0
0	5	2	94	91	90
		3	47	35	90
		4	123	121	90
		6	144	139	90
		7	15	21	90
		8	67	74	90
		10	69	64	90
		11	14	16	270
0	6	0	41	45	180
		1	47	48	180
		2	7	14	0
		3	65	63	180
		4	7	17	0
		5	154	157	180
		7	160	118	180
		8	12	13	0
		9	70	78	180
		11	14	11	180
0	7	1	31	33	90
		2	59	59	270
		3	103	84	270
		5	9	5	90
		6	82	81	270
		7	16	14	90
		8	80	90	270
		9	22	23	90
		10	57	59	270
		11	29	33	270
0	8	0	58	60	180
		1	88	110	0
		2	80	90	180
		3	14	14	0
		4	20	7	0
		5	31	34	0
		6	50	50	180
		7	41	48	0
		8	12	13	0
		9	47	48	0
		10	28	32	0
		11	20	34	0
0	9	1	133	146	270
		2	42	41	270
		3	127	138	270
		4	26	24	90
		5	74	73	270
		6	51	59	90
		7	26	27	90
		8	40	45	90
		9	13	34	90
		11	15	14	90
0	10	0	201	214	0
		1	55	60	180
		2	80	139	0
		3	60	58	180
		4	154	141	0
		5	59	64	180
		7	17	9	180
		8	18	19	180
		9	43	40	180
		10	35	37	180
		11	15	12	180
0	11	1	141	140	90
		2	60	67	270
		3	99	115	90
		4	23	24	270
		5	34	33	90
		6	58	58	270
		7	12	12	90
		8	13	8	270
		9	24	27	270
		11	15	20	270
0	12	0	29	30	180
		1	75	66	180
		2	100	115	180
		3	45	43	0
		4	43	52	180
		5	34	34	180
		10	29	23	0
		11	13	14	180
0	13	1	118	107	270
		2	74	84	270
		3	33	40	270
		4	35	34	270

Column group 2

h	k	l	F_o	F_c	$\alpha°$
		5	28	32	270
		6	50	48	270
		7	13	16	270
		8	43	46	270
		10	21	21	270
		11	41	47	90
0	14	0	102	92	0
		1	29	28	180
		2	70	75	0
		3	72	77	0
		4	55	59	0
		5	69	82	0
		6	38	43	0
		9	38	43	180
		10	25	28	180
0	15	1	72	70	90
		2	46	46	90
		3	30	30	90
		4	68	76	90
		6	76	76	90
		8	37	42	90
		10	25	29	90
0	16	0	50	50	180
		1	41	38	180
		2	26	27	180
		3	60	61	180
		5	62	66	180
		7	14	21	0
		9	42	42	180
0	17	2	21	8	270
		4	61	66	270
		5	13	13	270
		6	65	71	270
		8	46	52	270
		9	15	21	90
		10	15	31	270
0	18	0	21	27	180
		1	51	47	0
		2	28	21	180
		3	53	48	0
		4	19	18	180
		5	36	39	0
		7	20	31	0
		8	15	22	0
		9	33	34	0
		10	14	13	0
0	19	1	57	56	270
		2	13	15	90
		3	76	64	270
		4	33	35	90
		5	14	17	270
		6	45	50	90
		8	25	36	90
		9	15	30	90
0	20	1	160	128	0
		2	30	30	180
		3	69	54	0
		4	14	13	180
		5	61	61	180
		6	14	12	0
		7	39	49	180
		9	14	18	180
0	21	1	61	51	90
		2	14	11	270
		3	50	53	90
		4	59	52	270
		5	25	19	90
		6	15	19	270
		7	25	19	90
0	22	0	58	46	180
		1	28	27	180
		2	59	46	180
		3	29	26	0
		4	38	39	180
		5	15	15	180
0	23	1	59	55	270
		2	20	18	90
		3	46	43	270
		4	35	21	270
		5	21	27	270
0	24	1	60	58	0
		2	46	37	0
		3	39	32	0
		4	46	44	0
0	25	1	53	50	180
		2	46	33	90
		3	37	29	90
0	26	0	53	52	180
		2	21	14	180
		3	14	16	180
0	27	1	20	17	270
0	28	0	11	1	180
0	29	2	14	15	90
0	30	0	16	19	0
0	31	1	18	21	90
0	32	0	16	17	180
0	33	2	17	19	180
0	34	1	17	26	270
0	35	2	24	29	0
1	0	0	172	179	90
		1	15	10	180
		2	155	153	180
		3	60	59	90
		4	94	96	180
		5	39	40	90
		6	72	67	90
		7	24	37	180
		8	24	16	180
		9	20	27	90
		10	19	15	0
		11	18	15	0
1	1	1	124	114	210
		2	139	132	102
		3	105	105	279
		5	110	93	105
		7	97	85	265
		9	42	40	249
		10	105	99	278
		8	14	22	200
		9	69	72	270
		10	29	34	177

Column group 3

h	k	l	F_o	F_c	$\alpha°$
		11	25	29	275
1	2	0	14	17	90
		1	232	188	141
		2	162	158	353
		3	96	73	182
		4	96	73	333
		5	18	8	203
		6	158	136	21
		7	34	33	226
		8	69	70	346
		9	44	41	0
		10	37	43	10
		11	31	26	44
1	3	0	43	51	270
		1	80	86	260
		2	188	169	269
		3	121	132	119
		4	82	74	271
		5	86	84	96
		6	24	18	347
		7	96	90	104
		8	20	30	120
		9	47	49	73
		10	17	18	81
		11	25	19	80
1	4	0	42	53	270
		1	162	167	351
		2	101	98	222
		3	103	77	38
		4	64	61	173
		5	72	60	354
		6	34	26	170
		7	13	21	110
		8	38	36	187
		9	27	15	182
		10	17	16	190
		11	31	33	164
1	5	0	94	123	90
		1	76	67	52
		2	128	136	115
		3	46	38	66
		4	87	86	92
		5	35	33	308
		6	55	51	68
		7	49	45	263
		8	21	20	262
		10	38	36	256
		11	25	19	203
1	6	0	22	19	90
		1	174	173	166
1	6				
		1	49	46	102
		2	76	71	190
		3	66	51	162
		4	66	57	162
		5	14	11	264
		7	33	30	213
		8	16	26	29
		9	24	27	320
		10	36	40	19
1	7	0	88	97	270
		1	63	70	204
		2	134	138	281
		3	92	80	245
		4	24	29	299
		5	41	35	294
		6	58	53	273
		7	26	28	99
		9	16	24	234
		10	35	38	103
		11	16	16	277
1	8	1	124	150	348
		2	19	25	9
		3	125	106	353
		5	50	56	11
		6	27	31	57
			76	74	353
		8	14	19	341
		9	44	43	5
		10	33	30	187
		11	25	32	357
			26	30	182
1	9	0	117	121	90
		1	22	24	195
		2	76	88	70
		3	24	49	114
		4	48	52	55
		5	83	77	79
		6	33	47	97
		7	16	15	338
		8	53	57	87
		9	17	24	288
		10	27	23	125
1	10	0	126	181	0
		1	38	40	161
		2	27	31	181
		3	23	36	145
		4	65	79	167
		5	67	62	112
		6	62	63	185
		7	40	40	76
		8	66	74	177
		9	53	42	90
		10	46	44	197
1	11	1	24	23	237
		2	58	68	272
		3	44	44	137
		4	52	55	270
		5	69	79	269
		6	52	55	270
		7	19	42	167
		8	58	67	341
		9	18	26	222
		10	98	96	6
		11	20	20	98
			98	100	357
1	12	1	16	19	188
		2	13	55	15
		3	57	26	270
		4	60	51	145
		5	58	45	251
		6	59	67	92
1	13	1	31	27	276

Column group 4

h	k	l	F_o	F_c	$\alpha°$
		-5	83	76	95
		7	82	81	85
		9	31	37	82
1	14	1	75	80	19
		2	64	67	207
		3	19	29	28
		4	45	51	193
		5	72	59	3
		6	39	41	182
		8	35	37	177
1	15	0	100	77	90
		1	14	20	316
		2	92	93	78
		3	25	28	318
		4	21	33	108
		5	27	36	288
		6	15	25	116
		7	29	35	255
		9	20	33	90
1	16	0	95	89	184
		1	15	20	92
		2	71	77	176
		4	22	25	48
		5	16	19	197
		6	16	13	72
		7	85	89	270
1	17	1	15	15	189
		2	100	91	269
		3	22	19	163
		4	42	45	281
1	18	0	22	20	270
		1	75	82	12
		2	22	23	354
		3	66	66	4
		4	16	21	55
		5	34	36	351
		6	77	75	90
		7	59	55	81
1	19	1	17	16	96
		2	42	45	78
		3	35	38	74
		4	34	34	176
		5	45	46	144
		6	49	41	147
		7	18	21	141
		8	25	30	182
1	20	0	30	25	270
		3	40	47	291
		4	26	27	265
		5	12	15	337
		6	40	48	341
		7	31	36	31
		8	45	51	355
1	20		20	25	270
		5	30	25	270
		6	15	30	182
1	21	0	40	47	291
		3	43	44	188
		4	20	25	157
1	22	1	12	15	337
		2	40	48	341
		3	31	36	31
		4	45	51	355
1	23	0	20	25	270
		2	36	37	116
		3	45	45	84
		4	14	7	270
1	24	2	25	29	204
		3	18	22	1
		4	32	28	177
		5	28	25	30
1	25	0	29	29	90
		2	30	37	120
		3	31	29	266
		4	40	41	173
1	26	1	19	10	63
		2	31	30	270
		3	34	37	276
1	27	0	44	41	351
1	28	1	31	33	90
1	29	0	29	30	80
1	30	2	23	25	172
1	31	0	14	17	270
2	0	0	199	171	180
		1	17	1	90
		2	108	106	180
		3	56	61	90
		4	48	40	180
		6	44	44	0
		10	31	26	0
		11	10	27	58
2	1	0	109	105	277
		1	56	52	236
		2	135	132	243
		3	57	59	210
		4	64	71	291
		5	51	39	304
		6	11	14	146
		7	49	36	250
		8	41	42	116
		9	27	23	125
		10	126	181	0
2	2	0	83	60	300
		1	176	134	30
		2	57	42	337
		3	32	42	350
		4	107	93	327
		5	47	38	297
		6	33	38	16
2	2	11	10	19	350
2	3	0	70	74	0
		1	140	101	79
		2	44	50	49
		3	36	36	67
		4	38	41	89
		5	25	25	44
		6	105	70	78
		7	8	12	110
		8	57	46	65
		9	29	31	341
		10	14	29	114
		11	14	45	276
2	4	0	25	12	52
		1	26	27	270
		2	60	51	145
		3	58	53	136
		4	70	57	186
		5	123	113	190
		6	37	42	82

Column group 5

h	k	l	F_o	F_c	$\alpha°$
		7	106	85	173
		8	57	45	71
		9	38	41	206
		10	17	19	45
		11	46	24	121
2	5	0	51	50	180
		1	35	31	197
		2	129	84	277
		3	43	37	240
		4	131	98	260
		5	80	74	194
		6	64	75	265
		8	64	63	287
		10	51	60	263
		11	15	23	178
2	6	0	21	17	0
		1	57	48	337
		2	25	22	252
		3	56	67	2
		4	46	45	282
		5	92	85	5
		6	36	34	250
		7	60	68	14
		8	12	17	266
		9	34	44	352
		10	11	27	28
2	7	0	5	2	180
		1	38	49	290
		2	45	33	156
		3	44	40	277
		4	56	59	123
		5	40	36	337
		6	35	40	109
		7	14	10	67
		8	66	65	71
		9	10	22	12
		10	23	35	113
		11	15	6	109
2	8	0	73	68	0
2	8	1	121	124	220
		2	99	87	3
		3	65	62	202
		4	49	55	38
		5	53	47	191
		6	32	21	53
		7	49	58	189
		8	43	44	188
		10	20	25	157
2	9	0	30	26	0
		1	55	68	81
		2	61	56	355
		3	96	90	108
		4	84	73	289
		5	24	36	123
		6	59	54	262
		7	26	17	161
		8	42	48	264
		9	28	32	233
		10	20	20	195
2	10	0	152	153	180
		1	55	54	99
		2	43	50	203
		3	39	38	357
		4	53	53	185
		5	28	25	26
		7	23	21	345
		9	14	17	312
2	11	0	84	84	281
		1	11	11	180
		2	31	44	172
		3	86	93	260
		4	20	29	203
		5	36	39	276
		6	20	19	110
2	12	0	61	54	304
		1	63	78	347
		2	20	19	287
		3	64	68	346
		5	18	21	276
		7	15	18	327
		8	17	25	178
2	13	0	89	86	95
		1	8	8	34
		2	52	55	81
		3	49	45	34
		4	35	34	25
		5	16	25	90
		6	20	27	79
2	14	0	44	44	180
		1	8	18	107
		2	46	53	154
		3	37	42	166
		4	38	49	162
		5	34	32	102
		6	40	43	174
		7	25	24	43
2	14		23	17	278
2	15	0	36	30	180
		1	54	54	271
		2	55	44	251
		3	19	25	220
		4	53	53	262
		5	20	25	227
		6	34	39	258
		7	37	45	272
		8	12	22	108
2	16	0	56	49	0
		1	13	5	5
		2	40	47	342
		3	26	28	124
		4	34	42	354
		5	14	10	111
		6	29	43	2
		7	20	23	75
		8	23	35	6
2	17	0	48	42	180
		3	9	10	325
		4	42	51	104

Column group 6

h	k	l	F_o	F_c	$\alpha°$
		6	48	61	103
		10	5	5	338
2	18	0	18	31	92
		-8	18	31	92
		1	41	40	0
		1	41	37	201
		2	16	18	26
		3	48	46	223
		4	14	9	342
		5	45	58	197
		6	32	36	72
		7	31	36	177
		8	38	18	122
2	19	0	34	29	0
		1	34	32	100
		2	27	21	316
		3	35	29	123
		4	45	46	285
		5	14	30	168
		6	43	48	254
		7	10	1	141
		8	26	35	289
2	20	0	56	52	180
		1	10	13	28
		2	24	24	210
		3	32	36	27
		4	25	30	198
		5	18	25	279
2	21	0	67	51	279
		1	34	34	152
		2	37	34	254
		3	21	26	284
2	22	0	17	17	278
		1	18	17	278
		2	68	63	4
		3	23	19	278
2	23	0	27	27	2
		5	10	14	220
2	24	0	50	41	0
		5	15	19	63
		6	43	39	180
		7	36	36	161
2	25	0	10	8	180
		1	27	24	280
2	26	0	37	32	0
		2	21	13	342
		1	14	17	120
2	27	0	18	21	215
		1	14	17	302
2	28	1	18	21	215
2	29	2	14	17	302
2	30	0	14	17	180
2	31	1	18	15	287
2	32	2	15	15	0
		2	21	23	350
2	33	1	16	20	106
2	34	3	13	18	180
3	0	1	9	9	82
		2	53	46	90
		3	23	18	270
		5	48	42	180
3	1	0	37	37	270
		1	60	52	130
		2	35	36	36
		3	51	31	117
		4	86	46	281
		5	47	43	97
		6	47	38	37
		7	51	55	70
		8	37	43	81
		9	37	32	0
		10	25	39	32
3	2	0	44	55	270
		1	76	72	13
		2	42	37	253
		3	107	84	61
		4	116	77	168
		5	62	54	313
		6	32	29	135
		7	56	56	278
		8	28	23	239
		9	12	15	215
		10	57	51	178
3	3	0	21	38	90
		1	43	29	319
		2	99	79	99
		3	67	51	295
		4	77	85	145
		5	29	29	135
		6	56	56	278
		7	28	28	239
		8	12	23	132
		9	12	23	93
3	4	0	20	90	0
		1	54	57	178
		2	79	57	50
		3	59	68	206
		4	45	45	54
		5	22	16	196
		6	62	66	24
		7	31	31	269
		10	17	21	12
3	5	0	31	31	270
		5	55	55	193
		6	76	68	280
		7	45	45	272
		2	39	39	95
		3	70	59	151
		4	39	39	283
		5	36	26	135
		6	28	18	251
		7	11	12	72
		8	24	23	132
		9	12	23	93
3	6	0	63	70	270
		1	59	55	299
		2	38	46	330
		3	29	29	288
		4	40	29	350
		6	47	44	333
		8	23	15	179

TABLE 4. (*Continued.*)

```
 h  k  l   F_O F_C  α°   | h  k  l   F_O F_C  α°   | h  k  l   F_O F_C  α°   | h  k  l   F_O F_C  α°   | h  k  l   F_O F_C  α°   | h  k  l   F_O F_C  α°
       10  31  31  19    |        6  26  33 156    | 4  1  0  24  44   0    | 4 10  0  59  58   0    |       11  11  12 269    |        6  24  32 192
 3  7  0  98 107  90     |        7  54  60 271    |       1  42  41  85    |        1  39  39 270    |       12   9   4 352    |        8  11  12 181
       1  37  51   1     |        9  22  26 276    |       2  50  45 321    |        2  29  34   3    | 5  3  0   8   2  90     |        9  13   8 293
       2  86  85  90     | 3 14  1  42  35 171     |       3  42  36  90    |        3  40  38 239    |       1  16  29 167     | 5 12  1  17  20  67
       3  81  70  47     |       2  42  40  35     |       4  34  36   1    |        4  20  18  26    |       2  11   9 209     |       3  12  12 317
       4  68  57   8     |       4  31  30  16     |       5  35  11  97    |        5  15  25 232    |       3  22  25 126     |       4  14  14 359
       5   9   8  37     |       6  43  44  35     |       7  15   6 185    |        6  32  23 158    |       4  36  33 331     |       5  26  28 294
       6  24  27  55     |       7  12  20 225     |       9  17  31 318    | 4 11  0  47  54   0    |       8  13  13 349     | 5 13  1  11   6 184
       8  20  24 312     | 3 15  0  10  23 270     | 4  2  0  14  36 180    |       1  32  31  97    |      10  17  16  36     |       2  15  11 340
       9  32  33  91     |       1  38  37 209     |       1  10  31 120    |       2  19  27 323    |      11  11   8 350     |       3  16  23 152
      10  25  22 279     |       2  39  38 260     |       2  46  37  66    |       3  20  28  82    | 5  4  0  29  41 270     | 5 14  0  27  30 270
 3  8  0  58  54  90     |       3  19  36 175     |       4  40  38 157    | 4 12  1  14  31 105    |       1  28  30 351     |       1  12   8 299
       1  42  59 183     |       4  30  29 267     | 4  2  5  28  20 157    |       2  31  38 198    |       2  24  23 272     |       2  15  10 275
       2  42  41 110     |       5  23  27  94     |       6  39  29 121    |       3  25  23 129    |       3  13  18  74     |       3  12  21  41
       3  76  70 157     |       6  12  13 358     |       8  28  24  65    |       5  22  11  57    |       6  19  18 251     |       4  14  18 209
       4  28  26 119     |       7  18  21 130     |       9  17  20 196    |       6  28  27 115    |       9  11   6 155     | 5 15  0  17  20  90
       5  30  29 120     | 3 16  0  24  22 270     | 4  3  0  23  37 180    | 4 13  0  14  28 180    |      12   9  12  70     |       1  25  21 341
       6  42  35 167     |       1  60  60   4     |       1  29  37 245    |       1  25  33 246    | 5  5  0  20  27  90     |       2  15  15 102
       7  40  35  74     |       2  44  44 243     |       2  34  27 217    |       2  21  26 202    |       1  17  25 348     |       3  14  21 358
       8  37  39 235     |       3  11  26  16     |       3  35  32 203    |       3  23  23 216    |       2  37  34 109     | 5 16  1  15  12 171
       9  37  27   1     |       4  12  20 254     |       4  34  36 216    |       4  22  33 204    |       3  30  40   8     |       2  14  13  72
 3  9  0  80  81 270     |       5  43  43 343     |       5  33  35 228    | 4 14  0  33  26   0    |       4  15  18  46     |       3   9  12 159
       1  28  37 164     |       6  21  27 303     |       6  24  29 227    |       1  21  26 270    |       5  15  20 275     |       4  14  13  72
       2   8  24 194     |       7  13  18 185     |       7  26  26 174    |       2  15  28 330    |       7  13   7   4     |       5  12   4 296
       3  35  34 233     | 3 17  0  47  47  90     | 4  4  1  52  43 266    |       3  22  22 303    |       8  12  10 213     |       8  13  11 327
       4  29  29 237     |       1  47  41 338     |       2  40  33 342    |       4  29  29 298    | 5  6  0  17  26  90     |      10  10   7 284
       5  50  51 280     |       2  55  57  89     |       3  52  35 354    | 4 15  1  31  21  86    |       1  12  20 176     | 5 17  1  26  31 181
       6  23  21 154     |       3  20  24  26     |       4  36  31 290    |       2  32  28  67    |       2  30  36  60     |       5  12  12 119
       7  22  29 287     |       4  32  34  68     |       5  46  43 332    |       3  23  19  68    |       3  36  35 177     |       9  15   8  64
       8  12  16 200     | 3 18  0  29  30  90     |       6  28  21 256    | 4 16  0  32  18 180    |       4  23  17  73    | 5 18  0  13  25 270
 3  9 10  13  29 130     |       1  42  45 154     |       7  30  29 354    | 4 17  2  24  19 324    |       7  14  13  86     |       1  21  18 270
 3 10  0  29  22  90     |       2  17  26 105     |       8  32  30 269    | 4 18  0  20  13   0    |       8  13  12 296     |       2  12  11 235
       1  26  24 298     |       3  50  49 182     | 4  5  0  19   4   0    | 4 19  1  18  21 318    |       9  12   9 282     |       3  12   9 346
       2  15  33 342     | 3 19  0  44  46 270     |       1  31  25  73    |       2  21  21 197    |      10  14   6 347     |       9  13  10 235
       3  32  32 324     |       1  12  21 186     |       2  55  50  59    | 4 20  1  21   9  50    | 5  7  0   9  24 270     | 5 19  0  17  18  90
       4  55  54   2     |       2  12  15 248     |       3  26  17 336    | 4 21  0  25  22   0    |       1  12  29 168     |       1   9  10  58
       5  14  18 344     | 3 20  1  28  23   9     |       4  49  35 121    | 4 23  1  28  22 275    |       2  19  13 247     |       3  16  17   1
       6  30  35 335     |       3  25  23 340     |       5  41  34  20    | 4 24  0  16  15   0    |       3  11  18 201     |       4  13  12  28
       7  36  33 264     |       4  22  26 358     |       6  25  28 104    |       1  29  19 273    |       5  27  17 174     |       6  16  12 347
       8  43  44   4     | 3 21  1  25  25  89     |       7  15  25 350    |       2  26  24 327    |       6  13   9 245     | 5 20  1  12   9 134
       9  18  23 250     |       2  36  29  74     |       8  28  32  71    | 4 25  0  18  17   0    |       7  10   5 329     |       2  16  15 109
      10  13  26 349     | 3 22  0  22  15 270     | 4  6  0  29  15 323    | 4 27  2  18   1 276    |       8  15  19 114     |       3  17  17 147
 3 11  0  29  20 270     |       1  13  15 157     |       1  43  29 271    | 5  0  1  27  26 270    |      11  11   9 270     |       4  12   5 244
       1  44  34 140     |       2  12  27  91     |       2  52  36 115    |       2   9   8 180    | 5  8  0  11   9 270     | 5 20  7  12  14  64
       2  20  30  54     |       4  28  25 167     |       3  48  42 185    |       3  40  36  90    |       2  26  30 306     | 5 21  2  14  14 255
       3  34  32  94     | 3 23  1  31  30 269     |       5  41  43  65    |       4  23  24 180    |       3  11   9 345     | 5 22  5  13  15 169
       4  41  33 349     | 3 24  0  12   8  90     |       7  35  41 177    |       5  11  21  90    |       4  10  25 299     |       6  13  12 203
       5  50  54  88     | 3 25  0  18  24 270     |       9  30  20 270    |       6  16  28 180    |       5  24  21 270    | 5 23  9  13  10 235
       6  35  37  39     |       1  23  22 140     | 4  7  1  21  24 140    |       9  16   6  90    |       6  13  16 236     | 5 24  1  18  14 271
       7  48  49  66     | 3 26  0  15  16 270     |       2  33  24 328    |      11  10   7  90    | 5  9  0  19  12  90     | 5 25  3  18  12 327
       8  30  28 356     |       1  22  22 354     |       3  30  24 176    | 5  1  0   8  25  90    |       3  23  27  39     | 5 26  4  12   7  49
       9  18  24  76     |       2  19  22 271     |       4  36  34 306    |       1   7  14 343    |       4  10  17  47     | 6  0  1  14  15  37
      10  18  24   9     | 3 27  0  20  23  90     |       5  43  46 156    |       2  14  25 215    |       6  14  16 331    |       2  10   8  69
 3 12  0  29  24 270     |       1  19  19 341     |       6  44  40 276    |       3  19   8 298    |       8  15  18  13     |       3  11  13 329
       1  33  27  85     |       2  27  26  93     | 4  8  0  40  31 180    |       4  20  23 167    | 5 10  0   8   0 270     |       5  11  90
       2  21  28 192     | 3 28  0  17  21  90     |       1  40  35  39    |       5  21  22 317    |       1  11  11 209     |       6   0  14  26 180
       3  35  38  50     |       1  15  16 176     |       3  30  34  35    |       7  27  21 269    |       2  13  15 109     |       2  12  12   0
       4  40  51 199     | 3 29  0  19  26 270     |       4  13  27 236    |       8  15  18 181    |       3  26  32  96     | 6  2  0  10  12 180
       5  39  37  73     |       2  12   6 260     |       6  21  27 275    |      10  11  11 179    |       4  16  22 199    | 6  4  0   9  14 180
       6  51  50 168     | 3 30  1  11  11  90     |       7   5  36 320    |      13   7  14  90    |       7  15  22  95     | 6  5  2  21  14 271
       7  20  24  94     | 3 31  0   8   4  90     | 4  9  0  31  39 180    | 5  2  1  32  27 275   |       8  17  19 190     | 6  6  0   6   5   0
       8  25  28 148     | 3 32  0   7  65   0     |       1  31  36 275    | 5  2  2  13  18  69   |       9  18  11  62     | 6  7  0  10   9   6
       9  22  21 136     | 4  0  1  35  53 270     |       2  39  40 181    |       3  18   7 155   |      11  12  11 321     | 6  8  0   8   3 180
 3 13  0  30  37  90     |       3  45  50   0     |       3  34  31 282    |       4   9  20  45   | 5 11  3  23  22 268     | 6 11  0  11  24 180
       1  10  10  47     |       5  51  36 270     |       4  14  13 173    | 5 11  5  23  22  20  |       5  18  23 314     | 6 12  0   9   7 180
       2  28  31  94     |       6  24   1 180     |       5  25  23 342    |       7  27  28 272   |                        |       7   4   4 180
       3  52  61 296     |       8  16   7 180     |       6  27  28  87    |                       |                        | 7  2  0   9  11 270
       4  26  36 180     |       9  24   9  90     |                        |                       |                        |
       5  27  23 273     |                        |                        |                       |                        |
```

The first three-dimensional Fourier synthesis was evaluated with the measured values of the structure amplitudes and the phase constants appropriate to the iodine atom alone. The approximately centrosymmetrical arrangement of the iodine atoms in the unit cell led to spurious planes of symmetry in the electron-density distribution and no clear details of the molecular structure could be discerned.

Improved co-ordinates were obtained for the iodine atom, and an electron-density distribution based on the revised phase angles was calculated; because of the spurious symmetry and high temperature factor the peaks were rather diffuse. Nine of the most prominent peaks were selected as atoms and were included (all as carbon atoms) in the calculation of a third set of structure factors. The value of R, the average discrepancy between calculated and observed structure amplitudes, fell from the previous value of 43% to $35 \cdot 7\%$.

A third electron-density distribution was then computed and co-ordinates were assigned to a further ten peaks. When structure factors were calculated on the basis of the iodine atom and nineteen carbon atoms, the value of R decreased to $34 \cdot 9\%$. The improvement was disappointingly small and it seemed that some of the peaks we had selected could not represent genuine atoms. We therefore plotted the co-ordinates of these nineteen peaks on a two-dimensional electron-density distribution based on the iodine phases. Five of the atoms fell on regions of rather low electron-density and were omitted from the next structure-factor calculation which was based, therefore, on the iodine atom and fourteen carbon atoms. The value of R was $33 \cdot 9\%$.

On examining the subsequent three-dimensional electron-density distribution it was realised that a rather distorted six-membered ring was not of the cyclohexane type but was actually a cyclopentane ring bearing a 1,2-epoxide. The biogenetic relation to limonin [4,7] (III) was

immediately evident and the location of the remaining atoms was straightforward.   The iodine atom, twenty-three carbon atoms, and five oxygen atoms were employed in the next calculation of structure amplitudes and phase constants, and the value of R fell to 29·6%.

In the next three-dimensional electron-density distribution all the atoms were clearly resolved and the correct chemical type could be assigned to each atom except in the furan ring where the choice of oxygen atom was still not unique.

Two further rounds of structure-factor and Fourier calculations were carried out.   Both F_o and F_c maps were computed, errors due to termination of series were corrected, and individual isotropic temperature factors were assigned.   The value of R was reduced to 20·4%.

The refinement was completed by performing four cycles of least-squares adjustment of the
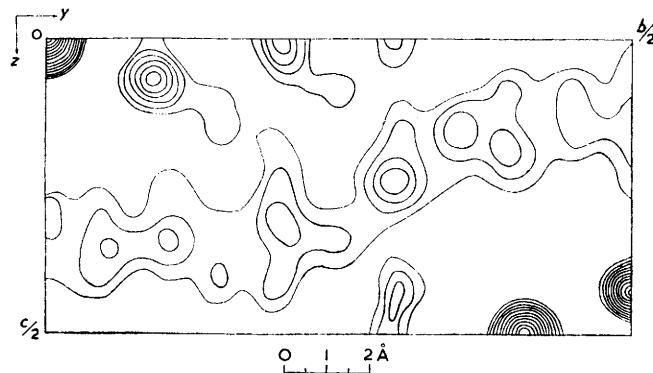
FIG. 5.   Patterson projection along the *a*-axis.   Contour scale arbitrary.
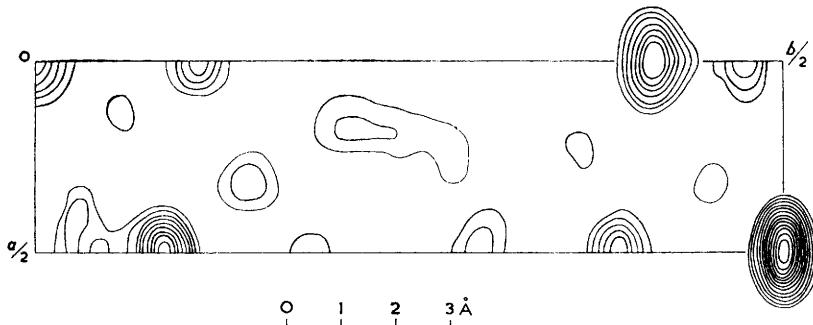
FIG. 6.   Patterson projection along the *c*-axis.   Contour scale arbitrary.

positional and thermal parameters of the atoms; the programme employed was that devised by Dr. J. S. Rollett.[20]   The final value for the discrepancy factor R is 17·5% over all the observed structure amplitudes.

During the refinement all the atoms of the furan ring were weighted as carbon atoms.   An examination of the bond lengths in the ring, the intermolecular contacts involving the ring, the temperature factors given by the least-squares refinement, and the peak heights led us to favour the oxygen assignment defined in Table 1 and Fig. 2.   However, the alternative assignment, C(23) an oxygen atom and O(5) a carbon atom, cannot be completely excluded.   It is possible that there is some disorder associated with the orientation of the furan ring in the crystal, the alternative assignments of the oxygen atom representing orientations of the furan ring related by rotation through 180° about the single bond C(17)–C(20).

The theoretical atomic scattering factors employed in the structure-factor calculations were

[20] Rollett, in " Computing Methods and the Phase Problem in *X*-ray Crystal Analysis," ed. Pepinsky, Robertson, and Speakman, Pergamon Press, Oxford, 1961, p. 87.

those of Berghuis *et al.*[21] for carbon and oxygen, and the Thomas–Fermi values [22] for iodine. The final atomic co-ordinates are in Table 1, the molecular dimensions and some non-bonded distances in Table 2, and the final values of $|F_o|$, $|F_c|$, and α in Table 4.

The standard deviations of the final atomic co-ordinates were derived from the least-squares residuals by application of the equation

$$\sigma^2(x_i) = \sum w_j(\Delta F_j)^2 / [(n-s) \sum w_j(\partial F_j/\partial x_i)^2].$$

The results are listed in Table 3.

As some doubt was felt about the physical significance of the anisotropic temperature factors derived for the carbon and oxygen atoms, we have not listed these parameters here but give in Table 1 an equivalent isotropic temperature factor for each atom deduced from the anisotropic thermal parameters; the anisotropic temperature factors have been listed elsewhere by Grant and Hamilton.[23] An examination of the anisotropic parameters showed that the average apparent thermal vibration of the atoms is considerably greater in the *a*-direction ($\bar{B}_{11} = 7 \cdot 1$ Å^2) than in the *b*- or *c*-direction ($\bar{B}_{22} = 4 \cdot 9$ Å^2, $\bar{B}_{33} = 4 \cdot 3$ Å^2). The large value of $\bar{B}_{11}$ is undoubtedly connected with the rapid decline in intensity values with increasing $\sin \theta$ mentioned earlier.

The final three-dimensional electron-density distribution was evaluated on the basis of the phase constants in Table 4 and is shown in Fig. 1 by means of superimposed contour sections drawn parallel to (001). The corresponding atomic arrangement is illustrated in Fig. 2.

[21] Berghuis, Haanappel, Potters, Loopstra, MacGillavry, and Veenendaal, *Acta Cryst.*, 1955, **8**, 478.
[22] " Internationale Tabellen zur Bestimmung von Kristallstrukturen," Borntraeger, Berlin, 1935, Vol. II, p. 572.
[23] Hamilton, Ph.D. Thesis, University of Glasgow, 1962; Grant, Ph.D. Thesis, University of Glasgow, 1962.