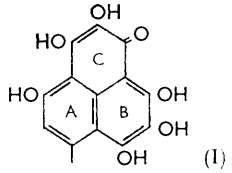
194. *Fungal Metabolites. Part III.*[1] *The Structure of Atrovenetin: X-Ray Analysis of Atrovenetin Orange Trimethyl Ether Ferrichloride.*[2]

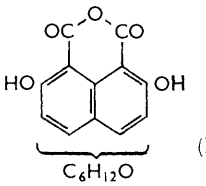
By I. C. Paul and G. A. Sim.

From the results of an *X*-ray study of atrovenetin orange trimethyl ether ferrichloride we assign the constitution (V) to atrovenetin, the metabolite of *Penicillium atrovenetum*. The crystals of the ferrichloride belong to the monoclinic system, space group $P2_1$–C_2^2, with four units of $C_{22}H_{25}O_6FeCl_4$ in the cell of dimensions $a = 17{\cdot}04$, $b = 9{\cdot}69$, $c = 15{\cdot}66$ Å, $\beta = 96°\ 35'$. Three-dimensional Patterson and Fourier methods were employed for the determination of the crystal structure. The two independent $C_{22}H_{25}O_6FeCl_4$ groups constituting the asymmetric crystal unit are disposed as if related closely, but not exactly, by a glide plane parallel to (010), and the majority of atoms in the unit cell conform approximately to the symmetry of the space group $P2_1/a$. This feature of the crystal structure made the refinement of atomic positions difficult. The average Fe–Cl bond length in the tetrahedral $FeCl_4^-$ anions is 2·17 Å.

Atrovenetin, $C_{19}H_{18}O_6$, was first isolated from *Penicillium atrovenetum* by Neill and Raistrick [3] who showed that the substance contains four hydroxyl groups (formation of monomethyl, trimethyl, and tetramethyl ethers), a hydrogen-bonded carbonyl group, and two or three *C*-methyl groups (Kühn–Roth). The tetramethyl ethers were insoluble in alkali and the remaining oxygen function was assumed to be ethereal.



(I)



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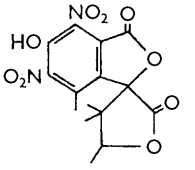

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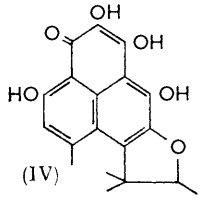

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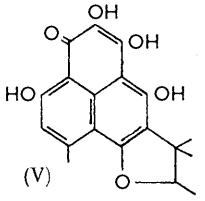

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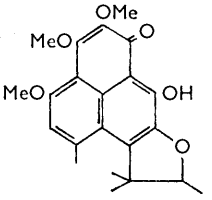

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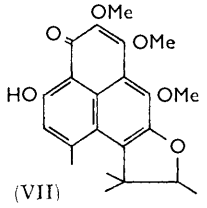
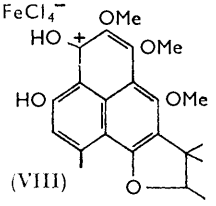
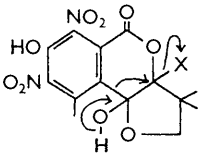


(VII)



(VIII)



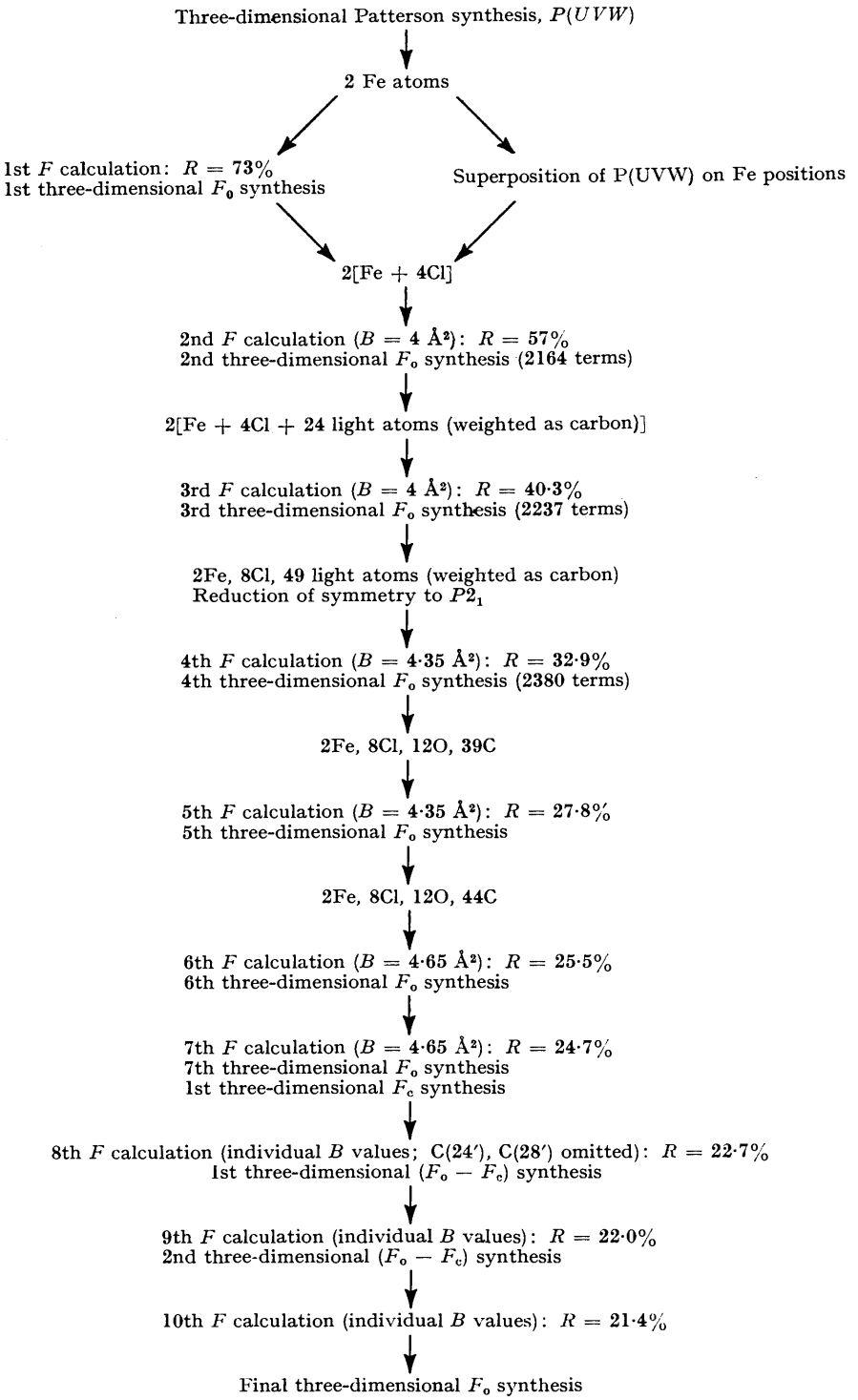
(IX)

The substances norherqueinone, $C_{19}H_{18}O_7$, and its monomethyl ether, herqueinone, $C_{20}H_{20}O_7$, are present as colouring pigments in *P. herquei* Bainier and Sartory,[4-6] a species

[1] Part II, Paul, Sim, Hamor, and Robertson, *J.*, 1963, 5502.
[2] For a preliminary report see Paul, Sim, and Morrison, *Proc. Chem. Soc.*, 1962, 352.
[3] Neill and Raistrick, *Biochem. J.*, 1957, **65**, 166.
[4] Stodola, Raper, and Fennel, *Nature*, 1951, **167**, 773.
[5] Galarraga, Neill, and Raistrick, *Biochem. J.*, 1955, **61**, 456.
[6] Harman, Cason, Stodola, and Adkins, *J. Org. Chem.*, 1955, **20**, 1260.

TABLE 1.

Progress of the structure analysis.



closely related morphologically to *P. atrovenetum*. Barton and his collaborators showed [7] that deoxynorherqueinone, obtained by reduction of norherqueinone with zinc and acetic acid,[5] is identical with atrovenetin. Acid hydrolysis of norherqueinone gives isopropyl methyl ketone and norxanthoherquein, $C_{14}H_{10}O_7$,[5,6] to which Barton, de Mayo, Morrison, and Raistrick [8] assigned structure (I) on the basis of spectroscopic and degradative studies. Oxidation of atrovenetin with alkaline hydrogen peroxide gave an optically active substance, $C_{18}H_{16}O_6$, of part structure (II) with hydrogen bonding between hydroxyl and carbonyl groups on both sides of the anhydride ring, proving that the ether bridge in atrovenetin is attached to ring B of the perinaphthenone nucleus.[8]

Neill and Raistrick obtained a phenol, $C_{15}H_{14}N_2O_9$, by oxidation of atrovenetin with nitric acid. Barton and his co-workers established the constitution (III) for this product and, as the ethereal oxygen is separated by two carbon atoms from the benzene ring in (III), deduced that (IV), rather than (V), must represent the constitution of atrovenetin.[8]

Atrovenetin forms two trimethyl ethers, one yellow and the other orange.[3] Barton and his collaborators showed by oxidative procedures that, on the basis of constitution (IV) for atrovenetin, the yellow and orange trimethyl ethers have to be allotted constitutions (VI) and (VII), respectively.[8]

TABLE 2.

Atomic co-ordinates and temperature factors.

(Origin of co-ordinates on two-fold screw axis.)

Atom	*x/a*	*y/b*	*z/c*	*B*	Atom	*x/a*	*y/b*	*z/c*	*B*
C(1)	0·3160	0·6686	0·3093	4·90	C(1′)	0·8152	0·3302	0·3052	4·90
C(2)	0·3075	0·6592	0·3895	4·55	C(2′)	0·8015	0·3373	0·3882	4·55
C(3)	0·3309	0·5455	0·4319	4·68	C(3′)	0·8243	0·4582	0·4410	4·68
C(4)	0·3928	0·3229	0·4426	4·72	C(4′)	0·8875	0·6894	0·4452	4·72
C(5)	0·4297	0·2084	0·3993	4·78	C(5′)	0·9356	0·7857	0·4089	4·42
C(6)	0·4427	0·2258	0·3095	4·42	C(6′)	0·9479	0·7753	0·3278	4·42
C(7)	0·4136	0·3378	0·1800	4·68	C(7′)	0·9152	0·6631	0·1828	4·68
C(8)	0·3911	0·4910	0·0405	4·42	C(8′)	0·8945	0·5184	0·0356	4·48
C(9)	0·3382	0·6299	0·0312	4·42	C(9′)	0·8957	0·3461	0·0501	4·55
O(10)	0·3405	0·6705	0·1222	5·15	O(10′)	0·8440	0·3265	0·1220	5·15
C(11)	0·3621	0·5709	0·1796	4·90	C(11′)	0·8597	0·4410	0·1698	4·72
C(12)	0·3496	0·5619	0·2611	4·68	C(12′)	0·8463	0·4347	0·2606	4·48
C(13)	0·3785	0·4411	0·3053	4·42	C(13′)	0·8767	0·5519	0·3101	4·42
C(14)	0·3654	0·4413	0·3970	4·37	C(14′)	0·8634	0·5648	0·3955	4·42
C(15)	0·4127	0·3304	0·2692	4·68	C(15′)	0·9135	0·6659	0·2720	4·68
C(16)	0·3889	0·4498	0·1334	4·72	C(16′)	0·8925	0·5400	0·1291	4·72
C(17)	0·3231	0·4132	−0·0104	4·42	C(17′)	0·8492	0·6233	−0·0212	4·55
C(18)	0·4613	0·4941	0·0057	4·48	C(18′)	0·9753	0·5699	−0·0004	4·48
C(19)	0·3540	0·7302	−0·0238	4·72	C(19′)	0·8558	0·2757	−0·0343	4·42
C(20)	0·2822	0·7997	0·2554	4·68	C(20′)	0·7785	0·2060	0·2528	4·72
O(21)	0·3198	0·5416	0·5203	5·25	O(21′)	0·8034	0·4638	0·5192	5·25
O(22)	0·3771	0·3028	0·5251	5·15	O(22′)	0·8759	0·6994	0·5245	5·15
O(23)	0·4586	0·0931	0·4573	5·15	O(23′)	0·9642	0·8854	0·4584	5·15
C(24)	0·4256	−0·0463	0·4310	4·42	C(24′)	0·9033	0·0024 *	0·4414	4·55
O(25)	0·4907	0·1336	0·2926	5·15	O(25′)	0·9878	0·8818	0·2848	4·90
C(26)	0·5636	0·1616	0·2753	4·68	C(26′)	0·0597 *	0·8688	0·3455	4·68
O(27)	0·4342	0·2315	0·1327	4·90	O(27′)	0·9365	0·7701	0·1381	4·90
C(28)	0·3824	0·1066	0·1218	4·42	C(28′)	0·8876	0·8859	0·1393	4·42
Fe	0·3717	0·1825	0·7499	4·75	Fe′	0·8697	0·8253	0·7489	4·75
Cl(1)	0·3898	0·0318	0·6501	6·00	Cl(1′)	0·8814	0·9599	0·6405	6·00
Cl(2)	0·3345	0·0602	0·8856	6·00	Cl(2′)	0·8346	0·9383	0·8535	6·00
Cl(3)	0·2792	0·3307	0·7045	5·50	Cl(3′)	0·7771	0·6685	0·7022	5·50
Cl(4)	0·4781	0·2898	0·7789	6·00	Cl(4′)	0·9767	0·7126	0·7797	5·75

* It is necessary to add a full unit cell translation to these co-ordinates to obtain a complete molecule.

[7] Barton, de Mayo, Morrison, Schaeppi, and Raistrick, *Chem. and Ind.*, 1956, 552.
[8] Barton, de Mayo, Morrison, and Raistrick, *Tetrahedron*, 1959, **6**, 48.

The argument for the orientation of the ether ring in atrovenetin being as in (IV) is invalid if a skeletal rearrangement occurs during the nitric acid degradation of atro-

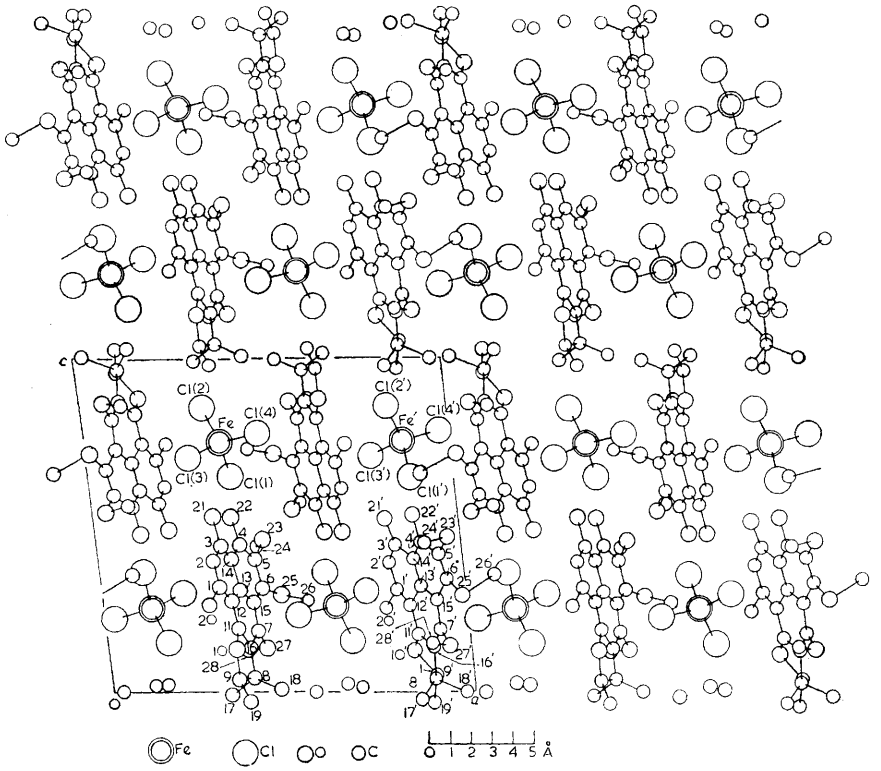


FIG. 1. The arrangement of molecules in the crystal as viewed in projection along the *b*-axis.

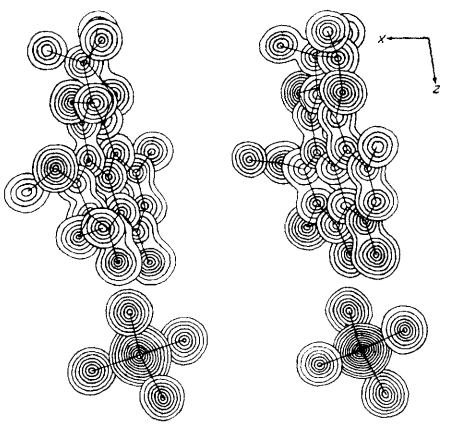


FIG. 2. The final three-dimensional electron-density distribution for atrovenetin orange trimethyl ether ferrichloride shown by means of superimposed contour sections drawn parallel to (010). Contour interval 1eÅ^{-3}, starting at the two-electron line, except around the iron and chlorine atoms where the interval is 3eÅ^{-3}.

venetin. The possibility of such a rearrangement was suggested by Professor R. B. Woodward and it therefore appeared desirable to determine the molecular structure of atrovenetin by an *X*-ray crystal-structure analysis of an atrovenetin derivative. Accordingly, Professor D. H. R. Barton and Dr. G. A. Morrison very kindly made available to us

crystals of the ferrichloride salts of the yellow and orange trimethyl ethers. Preliminary diffraction photographs of these derivatives showed that more extensive *X*-ray data could be recorded for the orange salt and we chose it for detailed study.

By employing the Patterson superposition method [9] followed by the usual phase-determining heavy-atom method [10] we were able to effect a direct determination of the crystal structure. The crystals are monoclinic, space group $P2_1$, with four $C_{22}H_{25}O_6FeCl_4$ groups in the unit cell. The asymmetric crystal unit consists of two $C_{22}H_{25}O_6FeCl_4$ groups which, though formally independent, are arranged as if related closely, but not exactly, by a glide plane perpendicular to *b* and a majority of the atoms in the unit cell conforms approximately to the symmetry of the space group $P2_1/a$. This feature helped the rapid elucidation of the chemical structure but severely hindered the refinement of atomic positions (cf. the *X*-ray analysis of cephalosporin C by Hodgkin and Maslen [11]). The progress of the analysis is summarized in Table 1.

Our results define the constitution of the ferrichloride as (VIII) and atrovenetin has therefore to be assigned structure (V) with the orientation of the ether ring opposite to that suggested earlier. The skeletal rearrangement necessarily involved in the formation of the phenolic degradation product (III) may proceed through an intermediate (IX), as indicated.

The arrangement of the molecules in the crystal as viewed in projection along the *b*-axis is shown in Fig. 1. The effect of the pseudo-glide is clearly visible in this Figure. The final three-dimensional electron-density distribution is shown in Fig. 2 as superimposed contour sections drawn parallel to (010) and covering the region of the two molecules in the asymmetric crystal unit; the corresponding atomic arrangement is explained in Fig. 1. Up to O(10) the numbering system is that given by Patterson, Capell, and Walker [12] for 7*H*-phenaleno-[2,1-*d*]-oxazole. After O(10) the numbering is arbitrary.

The greatest deviations from positions related by the pseudo-glide occur at C(9), C(17), C(18), C(24), C(26), and C(28). The ferrichloride anions and the planar portions of the organic cations, on the other hand, show only slight deviations. The difficulty of determining the proper displacements from the higher symmetry accounts for the rather high final value of the average discrepancy between measured and calculated structure amplitudes ($R = 21{\cdot}4\%$) and necessarily severely limits the accuracy with which the atomic co-ordinates (see Table 2) and the various interatomic distances and valency angles (see Table 3) have been determined. Theoretical and practical aspects of the problems inherent in the refinement of a pseudo-symmetric crystal structure have been discussed by Srinivasan [13] and Rae and Maslen.[14] There appears to be no completely satisfactory procedure.

It is not easy to obtain a true estimate of the errors in the various atomic co-ordinates. Standard accuracy calculations [15] are not strictly valid in the present case and have therefore not been carried out. A more realistic idea of the errors may be obtained by comparing measurements of corresponding bonds and valency angles in the two molecules (see Table 3). The overall root-mean-square deviations are 0·11 Å for the bond lengths and 11° for the bond angles. For the averaged dimensions we may divide by $\sqrt{2}$ and give 0·08 Å and 8° as reasonable estimates of the standard deviations.

The average aromatic carbon–carbon bond length in the atrovenetin molecules is 1·40 Å with a root-mean-square deviation of 0·06 Å. Because of the pseudo-symmetry the other

[9] Robertson and Beevers, *Acta Cryst.*, 1951, **4**, 270.
[10] 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.
[11] Hodgkin and Maslen, *Biochem. J.*, 1961, **79**, 393.
[12] Patterson, Capell, and Walker, " The Ring Index," 2nd edn., 1960, p. 589.
[13] Srinivasan, *Acta Cryst.*, 1961, **14**, 1163.
[14] Rae and Maslen, *Acta Cryst.*, 1963, **16**, 703.
[15] Cruickshank, *Acta Cryst.*, 1949, **2**, 65.

TABLE 3.

Interatomic distances (Å) and angles.

Intramolecular bonded distances

	1st molecule	2nd molecule	Average		1st molecule	2nd molecule	Average
C(1)–C(2)	1·28	1·35	1·32	C(8)–C(17)	1·52	1·50	1·51
C(1)–C(12)	1·44	1·37	1·40	C(8)–C(18)	1·37	1·63	1·50
C(1)–C(20)	1·59	1·55	1·57	C(9)–O(10)	1·47	1·52	1·50
C(2)–C(3)	1·32	1·46	1·39	C(9)–C(19)	1·35	1·57	1·46
C(3)–C(14)	1·32	1·46	1·39	O(10)–C(11)	1·34	1·35	1·35
C(3)–O(21)	1·42	1·32	1·37	C(11)–C(12)	1·32	1·47	1·40
C(4)–C(5)	1·48	1·41	1·45	C(11)–C(16)	1·48	1·31	1·40
C(4)–C(14)	1·40	1·47	1·44	C(12)–C(13)	1·42	1·44	1·43
C(4)–O(22)	1·36	1·28	1·32	C(13)–C(14)	1·48	1·39	1·44
C(5)–C(6)	1·46	1·32	1·39	C(13)–C(15)	1·37	1·44	1·41
C(5)–O(23)	1·49	1·30	1·40	O(23)–C(24)	1·50	1·54	1·52
C(6)–C(15)	1·27	1·45	1·36	O(25)–C(26)	1·33	1·46	1·40
C(6)–O(25)	1·26	1·45	1·36	O(27)–C(28)	1·50	1·41	1·46
C(7)–C(15)	1·40	1·40	1·40	Fe–Cl(1)	2·19	2·17	2·18
C(7)–C(16)	1·35	1·48	1·42	Fe–Cl(2)	2·19	2·12	2·16
C(7)–O(27)	1·34	1·38	1·36	Fe–Cl(3)	2·19	2·25	2·22
C(8)–C(9)	1·62	1·68	1·65	Fe–Cl(4)	2·09	2·13	2·11
C(8)–C(16)	1·51	1·48	1·50				

Intramolecular non-bonded distances

	1st molecule	2nd molecule		1st molecule	2nd molecule
C(4) · · · C(24)	3·63	3·05	C(17) · · · C(28)	3·69	3·58
C(5) · · · C(26)	3·20	2·57	C(18) · · · C(19)	2·93	3·51
C(6) · · · C(24)	3·28	2·98	C(18) · · · O(27)	3·30	2·97
C(6) · · · C(28)	3·21	3·19	C(18) · · · C(28)	4·45	4·15
C(7) · · · C(26)	3·28	3·88	O(22) · · · C(24)	3·82	3·27
C(8) · · · O(27)	2·95	2·91	O(23) · · · C(26)	3·60	2·55
C(8) · · · C(28)	3·94	3·92	O(25) · · · C(24)	3·09	3·21
C(15) · · C(26)	3·04	3·27	C(24) · · · C(26)	4·12	3·47
C(15) · · C(28)	3·16	2·97	O(25) · · · C(28)	3·07	2·68
C(16) · · C(28)	3·33	3·36	C(26) · · · O(27)	3·02	3·85
C(17) · · C(19)	3·13	3·38	C(26) · · · C(28)	3·72	4·07
C(17) · · O(27)	3·27	3·02			

Intermolecular distances (<4 Å)

O(22′) · · · Cl(1′)	3·10	Cl(2′) · · · C(12)$_{II}$	3·63	C(26) · · · C(20′)	3·75	C(4) · · · O(23)$_{II}$	3·84
C(24′) · · · O(21′)$_{I}$	3·10	C(24) · · · Cl(1)	3·64	Cl(4) · · · C(26)$_{II}$	3·75	C(14) · · · O(23)$_{II}$	3·84
C(24′) · · · Cl(1′)	3·21	O(22) · · · C(24)$_{II}$	3·65	C(2′) · · · Cl(1)$_{II}$	3·75	O(10′) · · · Cl(2)$_{II}$	3·84
O(22) · · · Cl(1)	3·27	C(19) · · · O(10′)$_{V}$	3·66	C(14′) · · · Cl(3)$_{II}$	3·75	Cl(2′) · · · C(16)$_{II}$	3·84
O(23′) · · · Cl(1′)	3·41	O(21) · · · Cl(3)	3·67	Cl(1′) · · · C(3)$_{II}$	3·75	Cl(3) · · · C(20′)$_{II}$	3·85
O(23) · · · Cl(1)	3·42	Cl(3′) · · · C(15)$_{II}$	3·67	C(20) · · · C(24)$_{IV}$	3·76	Cl(2′) · · · C(17)$_{II}$	3·86
O(22′) · · · Cl(3′)	3·43	C(18) · · · C(28)$_{V}$	3·68	C(24′) · · · C(3′)$_{I}$	3·77	C(2) · · · O(21′)$_{II}$	3·87
C(11) · · · Cl(4)$_{II}$	3·45	C(4) · · · C(24)$_{V}$	3·69	Cl(3′) · · · C(4)$_{II}$	3·77	C(6′) · · · Cl(3)$_{II}$	3·88
O(22) · · · Cl(3)	3·45	C(15′) · · · Cl(3)$_{V}$	3·71	Cl(3′) · · · C(6)$_{II}$	3·77	C(24′) · · · O(22′)$_{II}$	3·88
O(10) · · · Cl(4)$_{II}$	3·48	Cl(2′) · · · C(19′)$_{VII}$	3·71	O(21) · · · O(23)$_{II}$	3·78	C(24′) · · · Cl(4′)$_{II}$	3·88
C(11′) · · · Cl(2)$_{II}$	3·48	Cl(3′) · · · C(5)$_{II}$	3·71	C(13′) · · · Cl(3)$_{II}$	3·78	O(21) · · · C(2′)$_{II}$	3·90
C(18′) · · · C(19′)$_{III}$	3·49	Cl(4′) · · · C(18′)$_{VI}$	3·71	C(12) · · · Cl(4)$_{II}$	3·79	C(5′) · · · Cl(3)$_{II}$	3·90
C(2) · · · · C(24)$_{IV}$	3·51	Fe · · · · O(22)	3·72	C(20) · · · Cl(3′)$_{II}$	3·79	C(16′) · · · Cl(2)$_{II}$	3·91
O(21′) · · · Cl(3′)	3·56	C(4′) · · · Cl(3)$_{II}$	3·72	Cl(2′) · · · O(10)$_{II}$	3·79	C(28′) · · · C(17′)$_{V}$	3·91
Cl(4) · · · · O(25)$_{II}$	3·57	C(1) · · · C(24)$_{IV}$	3·73	Cl(3′) · · · C(14)$_{II}$	3·79	C(18) · · · Cl(2)$_{II}$	3·93
Cl(2′) · · · C(11)$_{II}$	3·57	Fe′ · · · · O(22′)	3·73	C(3) · · · O(23)$_{II}$	3·82	O(21) · · · C(24′)$_{VIII}$	3·94
C(9) · · · · C(19′)$_{V}$	3·60	Cl(1′) · · · C(2)$_{II}$	3·73	C(19) · · · C(19′)$_{V}$	3·82	C(20) · · · C(19′)$_{V}$	3·95
C(12′) · · · Cl(2)$_{II}$	3·60	Cl(3′) · · · C(13)$_{II}$	3·73	C(3′) · · · Cl(1)$_{II}$	3·82	O(22) · · · O(23)$_{II}$	3·95
O(10) · · · C(19′)$_{V}$	3·61	O(21) · · · C(26)$_{II}$	3·74	C(28′) · · · C(18′)$_{III}$	3·82	C(15) · · · Cl(1)$_{II}$	3·96
O(18′) · · · C(9′)$_{III}$	3·61	C(18) · · · O(27)$_{V}$	3·75	C(26) · · · Cl(1)$_{II}$	3·83	O(27′) · · · C(18′)$_{III}$	3·96
Cl(2′) · · · C(17′)$_{VI}$	3·62						

The subscripts refer to the following positions:

I	$2-x, \frac{1}{2}+y, 1-z$	IV	$x, 1+y, z$	VII	$x, 1+y, 1+z$
II	$1-x, \frac{1}{2}+y, 1-z$	V	$1-x, \frac{1}{2}+y, -z$	VIII	$1-x, -\frac{1}{2}+y, 1-z$
III	$2-x, \frac{1}{2}+y, -z$	VI	$x, y, 1+z$		

TABLE 3. (*Continued.*)

Valency angles

	1st molecule	2nd molecule	Average		1st molecule	2nd molecule	Average
C(2)C(1)C(12)	124°	125°	125°	C(9)O(10)C(11) ...	115°	102°	109°
C(2)C(1)C(20)	120	116	118	O(10)C(11)C(12)...	129	117	123
C(12)C(1)C(20) ...	116	117	117	O(10)C(11)C(16)...	109	114	112
C(1)C(2)C(3)	119	121	120	C(12)C(11)C(16)...	121	129	125
C(2)C(3)C(14)	124	113	119	C(1)C(12)C(11) ...	125	130	128
C(2)C(3)O(21)	116	118	117	C(1)C(12)C(13) ...	118	116	117
C(14)C(3)O(21) ...	120	128	124	C(11)C(12)C(13)...	116	114	115
C(5)C(4)C(14)	121	118	120	C(12)C(13)C(14)...	113	120	117
C(5)C(4)O(22)	118	120	119	C(12)C(13)C(15)...	126	122	124
C(14)C(4)O(22) ...	121	121	121	C(14)C(13)C(15)...	121	117	119
C(4)C(5)C(6)	119	121	120	C(3)C(14)C(4).....	124	116	120
C(4)C(5)O(23)	114	117	116	C(3)C(14)C(13) ...	122	123	123
C(6)C(5)O(23)	127	123	125	C(4)C(14)C(13) ...	114	121	118
C(5)C(6)C(15)	118	123	121	C(6)C(15)C(7).....	119	124	122
C(5)C(6)O(25)	107	122	115	C(6)C(15)C(13) ...	126	119	123
C(15)C(6)O(25) ...	134	115	125	C(7)C(15)C(13) ...	115	118	117
C(15)C(7)C(16) ...	123	123	123	C(7)C(16)C(8).....	134	130	132
C(15)C(7)O(27) ...	123	127	125	C(7)C(16)C(11) ...	118	114	116
C(16)C(7)O(27) ...	114	110	112	C(8)C(16)C(11) ...	108	116	112
C(9)C(8)C(16)	103	91	97	C(5)O(23)C(24) ...	115	104	110
C(9)C(8)C(17)	89	138	114	C(6)O(25)C(26) ...	123	92	108
C(9)C(8)C(18)	117	111	114	C(7)O(27)C(28) ...	120	110	115
C(16)C(8)C(17) ...	105	115	110	Cl(1)Fe Cl(2)	105	111	108
C(16)C(8)C(18) ...	121	115	118	Cl(1)Fe Cl(3)	111	106	109
C(17)C(8)C(18) ...	117	89	103	Cl(1)Fe Cl(4)	107	109	108
C(8)C(9)O(10)	101	103	102	Cl(2)Fe Cl(3)	110	110	110
C(8)C(9)C(19)	120	109	115	Cl(2)Fe Cl(4)	115	114	114
O(10)C(9)C(19) ...	116	110	113	Cl(3)Fe Cl(4)	109	106	108

carbon–carbon bond lengths are less reliable. The average sp^2-carbon–oxygen and sp^3-carbon–oxygen single bond lengths are 1·36 and 1·47 Å, respectively, in good agreement with values reported for other molecules.[16]

In the ferrichloride anions d(Fe–Cl) = 2·17 Å with r.m.s. deviation of 0·04 Å, while in tetraphenylarsonium ferrichloride the corresponding distance is 2·19 ± 0·03 Å.[17] On the basis of ionic radii the expected length of an ionic Fe^{III}–Cl bond (Fe^{III} in octahedral co-ordination) is about 2·46 Å, close to the value of 2·48 Å in solid ferric chloride;[18] with allowance for the contraction in ionic radius of iron on changing from octahedral to tetrahedral co-ordination, a value of about 2·4 Å should apply to the ferrichloride anion. It appears that covalent bonding may play an important role in the ferrichloride anion. A difference of about 0·2 Å between the sum of the ionic radii (2·56 Å) and the experimental Fe^{II}–Cl distance (2·38 Å) is also found in ferrous chloride tetrahydrate.[19]

The intermolecular approach distances (see Table 3) are all greater than 3 Å. It is possible that the contacts O(22) · · · Cl(1) and O(22′) · · · Cl(1′) represent hydrogen bonds; the distances involved are 3·27 and 3·10 Å, not appreciably different from values reported for OH · · · Cl hydrogen bonds in isoleucine hydrochloride monohydrate (3·07 Å)[20] and adenine hydrochloride (3·12 Å),[21] while the angles Cl(1)O(22)C(4) and Cl(1′)O(22′)C(4′) are 133 and 129°, respectively. The other intermolecular contacts correspond to normal van der Waals interactions.

[16] Cochran, *Acta Cryst.*, 1953, **6**, 260; Hassel and Viervoll, *Acta Chem. Scand.*, 1947, **1**, 149; Dunitz and Rollett, *Acta Cryst.*, 1956, **9**, 327; McCallum, Robertson, and Sim, *Nature*, 1959, **184**, 1863; Brown and Sim, *J.*, 1963, 1050.

[17] Zaslow and Rundle, *J. Phys. Chem.*, 1957, **61**, 490.

[18] Gregory, *J. Amer. Chem. Soc.*, 1951, **73**, 472.

[19] Penfold and Grigor, *Acta Cryst.*, 1959, **12**, 850.

[20] Trommel and Bijvoet, *Acta Cryst.*, 1954, **7**, 703.

[21] Broomhead, *Acta Cryst.*, 1948, **1**, 324; Cochran, *ibid.*, 1951, **4**, 81.

Experimental

Crystal Data.—Atrovenetin orange trimethyl ether ferrichloride, $C_{22}H_{25}O_6FeCl_4$; M = 582·8; m. p. 151—152°. Monoclinic, a = 17·04, b = 9·69, c = 15·66 Å, β = 96° 35′, U = 2669 Å^3, D_m = 1·526 g. cm.$^{-3}$ (by flotation), Z = 4, D_c = 1·510 g. cm.$^{-3}$, space group $P2_1$–C_2^2. Absorption coefficient for X-rays (λ = 1·542 Å) μ = 91 cm.$^{-1}$. Total number of electrons in the unit cell = $F(000)$ = 1196.

Experimental Measurements.—Rotation, oscillation, and Weissenberg photographs were taken with copper-K_α (λ = 1·542 Å) radiation and precession photographs were taken with molybdenum-K_α (λ = 0·7107 Å) radiation. The cell dimensions were derived from the precession photographs. For the intensity measurements small crystals, completely bathed in a uniform X-ray beam, were employed and no corrections for absorption were applied. The intensities were estimated visually from multiple-film equatorial and equi-inclination upper-layer Weissenberg photographs obtained from crystals rotated about the b-axis. The usual correction factors (Lorentz, polarization, and rotation [22]) were applied and 2360 independent

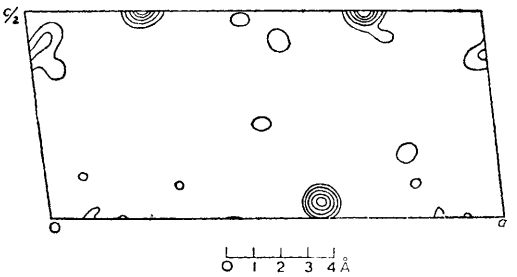


Fig. 3. Section through the three-dimensional Patterson synthesis at $V = \frac{1}{2}$.

structure amplitudes derived. The absolute scale of each layer of reflexions was obtained at a later stage by correlation of the $|F_o|$ values with the calculated structure amplitudes, $|F_c|$.

Analysis of the Structure.—There are four molecules of atrovenetin orange trimethyl ether ferrichloride in the unit cell. Since the space group $P2_1$ has two equivalent positions (x, y, z, and $\bar{x}$, $\frac{1}{2}+y$, $\bar{z}$) it follows that the asymmetric crystal unit consists of two molecules.

In the $h0l$ zone, reflexions with h odd are either absent or very weak, suggesting the presence of a pseudo-glide in the direction of the a-axis. As the atrovenetin molecule has an asymmetric centre at C(9) we assumed at this stage that the pseudo-glide probably only involved the ferrichloride anions. If the co-ordinates of one of the iron atoms in the asymmetric unit are (x, y, z) those of the atom related by the pseudo-glide are ($x+\frac{1}{2}$, $\bar{y}$, z). The vectors between the four iron atoms in the unit cell are then: (*a*) $\frac{1}{2}$, 2y, 0; (*b*) 2x, $\frac{1}{2}$, 2z; (*c*) $\frac{1}{2}+2x$, $\frac{1}{2}+2y$, 2z.

We were unable to locate the iron atoms unambiguously from an examination of the sharpened two-dimensional Patterson function and we therefore computed the three-dimensional Patterson function $P(UVW)$. The section at $V = \frac{1}{2}$ is shown in Fig. 3. A detailed consideration of this section, of the line $P(\frac{1}{2}, V, 0)$ which showed a large peak at about V = 9/24, and of the section $P(U, 3/24, W)$ enabled us to obtain the initial co-ordinates of the iron atoms as

	x/a	y/b	z/c
Fe(1)	0·374	0·179	0·751
Fe(2)	0·874	−0·179	0·751

In the application of the phase-determining heavy-atom method [10] the effectiveness of the heavy atom (or atoms) is usually measured by the ratio $\Sigma f_H^2/\Sigma f_L^2$, where f_H and f_L are the scattering factors of the heavy and light atoms, respectively.[23] The ratio of the square of the atomic number of iron to the sum of the squares of the atomic numbers of the chlorine, carbon, and oxygen atoms is 0·29. We considered, therefore, that calculations based solely on the iron atoms would give rather unreliable phase constants, $\alpha(hkl)$, and a subsequent electron-density distribution which would be difficult to interpret. In fact, when structure factors were calculated on the basis of the iron atoms alone the average discrepancy between calculated and observed structure amplitudes, R, was 73%.

[22] Tunell, *Amer. Min.*, 1939, **24**, 448.
[23] Sim, *Acta Cryst.*, 1957, **10**, 177, 536; see also ref. 10.

TABLE 4.

Measured and calculated values of the structure factors.

h	*k*	*l*	F_o	F_c	α
0	0	3	30	17	0
		4	108	100	0
		5	56	49	180
		6	10	3	180
		7	136	122	0
		8	78	80	0
		9	12	8	0
		10	50	50	180
		11	14	21	180
		12	18	21	180
		14	14	3	180
		16	14	8	0
		18	17	12	180
1	0	-11	8	6	0
		-10	11	10	180
		- 9	20	1	180
		- 7	21	16	180
		- 6	9	22	0
		- 5	11	6	0
		- 4	21	11	180
		- 3	7	9	0
		4	14	18	180
		5	23	10	0
		7	10	13	180
		8	12	3	180
		10	23	13	180
		11	17	5	180
2	0	-19	12	9	180
		-17	25	11	0
		-16	10	17	180
		-15	33	37	0
		-13	20	16	0
		-12	12	13	180
		-11	26	27	180
		-10	48	34	180
		- 9	136	139	0
		- 8	85	74	180
		- 7	55	53	180
		- 6	39	48	0
		- 5	89	80	0
		- 4	73	59	0
		- 3	160	167	180
		- 2	35	34	180
		2	24	15	0
		3	110	121	0
		4	40	22	180
		5	28	36	0
		6	60	70	180
		7	10	14	180
		8	44	49	0
		9	43	39	180
		10	22	19	0
		11	31	34	0
		13	19	23	180
		15	16	17	180
		17	10	5	180
3	0	-16	9	5	180
		-15	11	6	0
		-13	17	8	180
		-10	15	6	0
3	0	- 9	23	7	0
		- 8	9	7	0
		- 6	10	19	0
		- 4	10	2	180
		- 3	6	18	0
		- 2	7	1	0
		- 1	7	9	0
		1	25	16	0
		2	14	17	0
		3	5	2	0
		6	11	4	180
		7	20	10	0
		9	6	11	0
		10	14	5	0
		12	9	3	0
4	0	-18	22	12	0
		-16	24	19	180
		-15	13	12	180
		-14	31	28	0
		-13	26	33	0
		-12	9	11	180
		-11	7	10	180
		-10	91	92	0
		- 9	26	9	180
		- 8	127	128	180
		- 7	78	78	180
		- 6	92	97	0
		- 4	73	68	0
		- 3	35	21	180
		- 2	72	79	180
		- 1	74	77	180
		0	194	260	180
		1	25	27	0
		2	24	18	0
		3	89	87	0
		4	38	31	0
		5	11	11	180
		6	62	78	180
		7	24	37	0
		8	41	24	180
		9	32	28	0
		10	82	77	0
		12	33	36	180
		13	19	22	0
		14	18	25	0
5	0	-14	15	3	0
		-10	8	4	0
		- 8	48	10	180
		- 7	8	8	0
		- 6	22	16	180
		- 5	16	18	180
		- 4	9	9	180
		- 1	27	27	180
		0	6	4	180
		1	12	10	180
		2	7	8	180
		3	23	10	180
		4	18	11	0
		5	10	1	180
		8	12	10	0
5	0	9	13	6	0
		10	9	1	0
6	0	-13	14	13	180
		-12	13	13	180
		-11	29	26	0
		-10	15	19	0
		- 9	64	64	180
		- 8	69	62	0
		- 7	61	54	0
		- 5	12	15	180
		- 4	37	39	0
		- 3	40	33	180
		- 2	89	75	0
		- 1	37	24	0
		0	58	59	0
		1	65	69	0
		2	64	62	180
		3	32	48	180
		4	32	34	0
		5	46	48	0
		7	25	25	180
		9	64	68	0
		10	11	13	180
		11	36	49	180
		12	8	13	0
		13	30	42	0
7	0	- 9	44	3	180
		- 8	14	5	0
		- 7	15	23	0
		- 5	10	9	0
		- 4	7	11	0
		- 3	23	15	0
		- 2	10	3	180
		- 1	7	8	0
		1	25	3	180
		2	20	7	180
		4	21	1	180
		5	25	1	0
		6	14	13	180
		8	10	3	0
		10	13	9	180
8	0	-14	20	12	180
		-11	10	11	180
		-10	15	4	180
		- 9	33	37	0
		- 8	43	41	0
		- 7	51	52	180
		- 6	12	10	0
		- 5	23	26	0
		- 4	12	11	0
		- 3	23	20	0
		- 2	11	10	0
		- 1	42	29	0
		0	62	70	0
		1	12	16	180
		2	125	141	180
		3	23	32	0
		4	90	106	0
		6	68	72	180
		8	16	18	0
8	0	9	27	26	180
		10	33	45	180
		11	19	21	0
		12	12	12	0
		14	9	7	180
9	0	-11	15	6	0
		- 8	14	6	0
		- 6	11	9	0
		- 5	12	10	0
		- 3	8	0	0
		- 1	29	9	0
		1	15	4	180
		2	22	21	0
		4	13	3	0
		5	9	1	0
		9	17	9	180
10	0	-15	23	10	180
		-14	10	13	0
		-11	10	4	0
		-10	16	18	0
		- 8	44	43	180
		- 7	49	52	180
		- 6	17	21	0
		- 5	44	36	0
		- 3	45	37	180
		- 2	25	37	180
		- 1	70	46	0
		0	13	6	180
		1	85	107	180
		2	25	33	0
		3	72	68	0
		5	43	47	180
		7	12	6	0
		8	8	2	0
		9	8	8	180
		13	10	3	180
11	0	-11	10	10	0
		- 7	10	1	180
		- 3	14	14	180
		- 2	14	6	0
		0	24	13	180
		1	23	13	0
		2	10	3	180
		3	7	2	180
12	0	-12	18	13	180
		- 8	41	52	180
		- 6	44	43	0
		- 5	17	16	180
		- 4	81	72	180
		- 3	16	22	180
		- 2	30	30	0
		- 1	15	33	0
		0	34	41	180
		1	33	34	0
		3	28	25	180
		5	25	24	0
		6	8	10	180
		7	13	34	0
		8	11	12	0
13	0	- 8	13	11	0
13	0	- 6	18	2	0
		0	11	2	180
		5	10	2	0
14	0	-13	25	18	180
		-12	13	16	180
		-11	24	19	0
		-10	27	32	0
		- 9	37	48	180
		- 7	28	30	0
		- 6	18	20	180
		- 5	41	40	180
		- 3	28	27	0
		- 2	33	22	180
		0	18	1	180
		1	17	6	0
		2	33	39	180
		4	10	13	0
		5	12	22	0
		6	12	16	180
15	0	-10	16	2	0
		- 9	16	4	0
		- 5	15	7	0
		- 3	14	4	0
16	0	-11	9	3	180
		-10	12	14	0
		- 9	11	4	0
		- 5	18	23	0
		1	16	15	180
		5	16	25	180
		6	10	5	180
17	0	- 8	13	5	180
		- 6	11	1	0
18	0	-12	10	4	180
		-11	23	11	180
		- 9	15	18	0
		- 8	11	13	180
		- 6	15	15	0
		- 4	16	21	180
		- 3	10	4	180
		- 2	11	3	0
		- 1	11	12	0
20	0	- 4	13	11	180
		- 2	10	7	0
		0	12	13	180
		2	9	16	0
0	1	2	45	46	258
		3	41	36	286
		4	12	13	216
		5	86	85	95
		6	56	53	275
		7	53	41	83
		8	48	43	259
		9	62	48	274
		10	18	12	302
		11	46	45	101
		12	14	9	296
1	1	-12	15	19	187
		-11	16	14	179
		-10	25	24	341
		- 9	46	34	3
1	1	- 8	67	58	182
		- 7	76	80	192
		- 6	69	73	358
		- 5	99	97	356
		- 4	68	71	190
		- 3	64	79	182
		- 2	109	128	0
		2	48	40	351
		3	110	115	359
		4	11	18	193
		5	26	36	214
		6	55	56	174
		7	17	13	18
		8	22	33	348
		9	41	32	166
		10	22	5	97
		11	15	17	330
		13	24	23	180
		14	16	14	201
2	1	-16	14	6	282
		-14	22	20	270
		-12	12	11	253
		-11	15	7	61
		-10	41	48	96
		- 9	23	26	259
		- 8	55	45	278
		- 7	41	38	103
		- 6	31	34	102
		- 5	9	3	165
		- 4	133	125	91
		- 3	11	7	203
		- 2	78	69	87
		- 1	29	30	274
		0	59	78	96
		1	66	81	88
		2	62	66	101
		3	59	50	83
		4	37	34	120
		5	18	18	184
		6	19	10	257
		7	16	13	120
		8	37	32	248
		9	28	17	274
		10	63	64	89
		11	16	18	240
		12	16	17	266
		13	13	12	287
		14	16	13	258
3	1	-14	23	20	350
		-13	10	19	197
		-12	16	16	172
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		- 8	85	96	179
		- 7	27	22	359
		- 6	147	134	2
		- 5	45	35	171
		- 4	74	79	188
		- 3	103	95	11
3	1	- 2	85	88	5
		- 1	87	100	176
		0	143	202	179
		1	137	193	3
		2	17	13	359
		3	42	52	14
		4	34	27	339
		6	52	47	174
		7	30	22	211
		8	19	15	217
		9	25	23	38
		10	17	14	321
		12	16	15	163
		13	13	12	337
		15	27	25	178
4	1	-13	13	9	81
		-10	11	12	268
		- 9	47	42	263
		- 8	19	2	246
		- 7	10	7	135
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TABLE 4. (*Continued.*)

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		4	14	9	104
		5	26	18	79
		6	11	6	358
		8	39	36	92
		9	18	22	80
		10	24	21	258
		11	26	20	262
		12	13	9	83
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		-13	25	24	181
		-12	26	28	2
		-11	32	45	351
		-10	21	19	179
		- 9	42	49	181
		- 8	82	73	356
		- 7	28	18	172
		- 6	68	45	181
		- 5	26	23	175
		- 4	33	29	344
		- 3	20	6	216
		- 2	27	5	14
		- 1	89	70	349
		0	47	67	162
		1	102	116	190
		2	23	21	138
		3	8	15	153
		4	37	40	0
		5	29	39	18
		6	20	20	160
		7	14	25	211
5	1	8	20	18	11
		9	22	18	4
		10	46	61	184
		11	10	11	141
		12	24	39	359
		13	13	16	358
		14	15	16	178
6	1	-16	15	10	87
		-14	19	21	85
		-10	16	7	52
		- 9	21	15	90
		- 8	83	72	94
		- 7	54	50	270
		- 6	34	34	281
		- 5	17	21	259
		- 4	20	28	254
		- 3	57	67	89
		- 2	25	23	265
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		0	29	30	99
		1	48	57	270
		2	41	59	275
		3	18	18	75
		4	34	41	83
		5	14	24	58
		6	35	44	78
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		-12	27	10	299
		-11	16	13	178
		- 9	37	33	5
		- 8	21	19	242
		- 7	29	20	178
		- 6	22	11	219
		- 5	20	26	211
		- 4	51	52	188
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		5	41	47	161
		6	19	19	201
		7	48	47	1
		8	27	37	14
		9	46	54	187
		10	16	17	181
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		13	18	21	174
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		0	36	22	196
		1	32	44	262
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		3	19	28	127
		4	16	13	200
		5	14	27	88
		6	16	26	257
		7	14	16	262
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		-12	15	15	171
		- 8	25	32	176
		- 7	24	16	5
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		- 4	52	43	161
		- 3	44	41	176
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		1	21	19	134
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		4	48	67	186
		5	22	31	171
		6	34	28	341
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10	1	-11	18	21	254
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		- 8	25	34	266
		- 7	37	40	84
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		- 4	17	3	340
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		0	14	8	270
		1	40	36	83
		2	21	29	89
		3	32	31	246
		4	23	33	269
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		2	25	23	357
		3	41	48	178
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		5	33	36	347
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		0	19	6	349
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		0	14	14	346
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		- 8	13	2	41
		- 7	25	17	259
		0	14	8	111
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		5	30	24	358
		6	95	90	3
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		8	20	14	177
		9	12	9	150
		10	20	25	141
		11	19	24	357
		12	9	5	323
		13	9	8	188
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		4	13	14	82
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		6	10	15	52
		7	15	15	13
		8	14	8	344
		10	12	13	34
		11	14	24	25
		12	9	4	62
		13	23	17	21
		15	23	18	184
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3	2	- 6	12	16	277
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		- 3	27	7	345
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		0	76	94	92
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		2	93	105	355
		3	14	26	179
		4	49	40	352
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		6	37	31	176
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		9	22	17	346
		10	16	6	237
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		4	33	34	83
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		7	12	14	223
		8	21	24	100
		9	12	4	125
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		12	25	33	88
		13	7	7	162
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		- 4	9	16	213
		- 3	24	37	178
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		- 1	28	27	19
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		1	50	52	177
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		4	24	11	178
		5	36	55	177
		6	29	39	12
		7	45	59	7
		8	23	26	184
		9	40	50	176
		12	17	22	350
		13	19	24	183
		15	14	23	3
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TABLE 4. (*Continued.*)

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		0	90	82	271
		1	92	97	270
		2	108	130	88
		3	19	22	78
		4	35	31	260
7	2	5	13	16	335
		6	18	12	63
		7	25	22	254
		8	11	4	336
		9	14	11	52
		10	14	13	122
		11	10	9	315
		12	17	16	273
		13	8	3	113
		15	10	15	100
8	2	-14	7	4	357
		-13	19	17	180
		-12	32	25	167
		-11	9	9	48
		-10	27	23	1
		- 9	22	22	148
		- 8	12	15	335
		- 7	27	22	357
		- 6	18	19	176
		- 5	59	44	177
		- 4	57	66	183
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		0	12	2	174
		1	33	34	13
		2	29	35	171
		3	11	21	26
		4	82	102	178
		5	33	34	6
		6	52	70	10
		7	7	11	164
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		9	11	9	87
		10	8	11	35
		11	13	11	198
		15	10	3	327
9	2	-15	16	10	97
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		- 7	68	70	88
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		12	19	20	266
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		7	13	16	215
		8	19	18	352
		9	14	19	11
		10	9	4	307
		11	7	7	353
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11	2	-15	7	14	94
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		5	19	26	89
		6	9	10	243
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		8	13	11	103
		9	10	17	93
		10	13	16	286
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		3	14	9	177
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		9	16	16	109
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		13	23	31	90
		14	11	7	263
		15	32	31	277
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		5	53	52	4
		6	45	42	182
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		9	10	9	201
		10	7	5	284
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		4	78	84	94
		5	12	15	132
		6	85	92	272
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		8	65	68	92
		9	17	22	291
		10	26	29	267
		11	29	26	269
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		14	8	16	275
		15	8	5	22
		16	12	9	104
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		12	22	18	271
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TABLE 4. (*Continued.*)

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TABLE 4. (*Continued.*)

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		1	10	17	150
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		4	25	37	189
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		9	13	21	194
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		1	24	18	242
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		4	12	12	156
		5	20	24	352
		6	17	24	349
		7	25	35	187
		8	11	12	192
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		1	24	25	183
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TABLE 4. (*Continued.*)

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		3	30	34	357
		4	53	48	6
		5	21	23	194
		6	31	34	180
		8	8	3	36
		9	18	11	162
		10	12	5	234
		11	13	9	1
		14	10	9	181
1	6	-13	8	7	54
		-12	16	15	252
		-11	8	3	218
		-10	19	11	96
		- 9	21	19	282
		- 8	22	23	260
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		- 5	7	3	117
		- 4	21	20	96
		- 3	19	24	235
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		1	23	32	91
		2	9	7	49
		3	14	9	213
		4	37	39	96
		6	8	10	221
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		8	14	8	226
		9	31	40	91
		10	17	18	105
		11	10	9	256
		12	15	17	263
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		- 8	9	6	206
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		7	12	19	5
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		3	25	31	210
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		6	10	16	311
		7	27	40	180
		9	21	25	2
		11	18	20	174
		12	7	3	180
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		5	14	15	280
		6	8	11	291
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TABLE 4. (*Continued.*)

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		6	17	16	24
		7	10	11	19
		9	14	13	164
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		3	25	19	180
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		7	13	13	106
		10	6	7	228
		11	13	12	97
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		5	19	22	186
		7	20	15	3
		9	11	15	173
		11	12	15	1
		13	6	7	183
8	7	-11	10	6	264
		-10	7	6	265
		- 9	18	14	91
		- 7	8	12	78
		- 6	13	20	90
		- 5	10	2	298
		- 4	21	25	265
		- 3	9	13	280
		- 2	22	28	89
		- 1	16	11	303
		0	28	20	269
		1	12	13	71
		4	14	18	282
		5	12	16	281
9	7	- 8	14	14	186
		- 6	17	18	1
		- 3	22	27	186
		- 1	13	22	355
		0	20	21	192
		2	20	22	348
		3	13	16	359
9	7	4	23	28	198
10	7	- 3	8	10	93
		- 2	12	12	82
		- 1	6	7	134
		0	22	27	280
		1	9	8	282
		3	11	15	101
		4	11	11	65
		6	7	10	272
11	7	-10	7	10	356
		- 9	24	20	195
		- 7	5	3	19
		- 6	7	10	350
		- 5	12	13	206
		- 3	13	10	14
		- 1	21	15	192
		0	11	20	185
		1	21	24	6
		2	7	8	342
		3	14	20	182
		5	11	16	358
12	7	- 9	16	18	271
		- 5	9	2	68
		- 2	13	6	119
		- 1	5	5	167
		0	8	14	288
		2	7	12	114
13	7	- 8	8	9	355
		- 6	18	19	191
		- 5	10	5	208
		- 4	14	14	9
		- 3	10	9	7
		- 2	7	4	262
		- 1	6	9	177
		0	6	10	1
		2	11	12	183
14	7	- 5	6	9	250
		- 4	12	9	277
15	7	- 9	9	9	6
		- 7	11	10	174

Transposition of the origin of the three-dimensional Patterson function to the sites of the iron atoms gave us a superposition function [9] which clearly revealed four chlorine atoms grouped tetrahedrally around each iron atom and confirmed that the chlorine atoms associated with Fe(2) are related to those of Fe(1) by the pseudo-glide in the *a*-direction. Confirmation of the positions of the chlorine atoms was obtained by computing around the iron atoms a three-dimensional electron-density distribution based on the phase constants (signs) appropriate to the iron atoms; peaks which could be assigned confidently to the chlorine atoms were obtained.

The ratio of the sum of the squares of the atomic numbers of the iron and chlorine atoms to the sum of the squares of the atomic numbers of the carbon and oxygen atoms is 1·56 and phase constants calculated on the basis of the iron and chlorine atoms were therefore expected to be sufficiently accurate to yield an electron-density distribution in which the organic molecule would be discernible. Indeed, the structure-factor calculation led to a value of R of 57% and in the subsequent electron-density distribution the aromatic nucleus of the atrovenetin molecule was well defined. We had considered it possible that the pseudo-glide symmetry applied only to the ferrichloride anions and not to the organic molecules, but thorough study of the electron density revealed few elongated atomic peaks and it was clear that most of the carbon and oxygen atoms conform to within a few tenths of an Ångstrom unit to the pseudo-symmetry. Four atoms C(18), C(20), C(24), and C(28) could not be assigned co-ordinates but for the rest of the molecule the average peak height of an oxygen atom was about 4 e Å^{-3} while the carbon atoms had peak heights of 2·0—3·5 e Å^{-3}. At this stage it was already apparent that the earlier structure (IV) proposed for atrovenetin required to be revised to (V).

Twenty-four carbon and oxygen atoms were included with the iron and chlorine atoms in the calculation of a further set of structure factors. The value of R was reduced to 40% and in the subsequent electron-density distribution the remaining four carbon atoms were located.

So far in the analysis we had taken the full symmetry of $P2_1/a$ to apply to the contents of

the unit cell and the asymmetric unit consisted, therefore, of one ferrichloride anion and one atrovenetin molecule. The true space group, however, is $P2_1$ and the glide plane can be only approximate. There is, unfortunately, no direct way of establishing the exact deviation of each atom from its pseudo-symmetrical position. We decided to try to reduce the symmetry by calculating structure factors with C(19) omitted from one molecule and C(17′), C(18′), C(19′), C(24′), C(26′), and C(28′) omitted from the other molecule. The value of R was 32·9%.

In the subsequent electron-density distribution atoms C(24′), C(26′), C(28′) had rather lower peak heights than the other atoms and were omitted from the next structure-factor calculation. Atoms C(9) and C(9′) had peaks which were distinctly elongated in the x-direction and they too were omitted from the structure-factor calculation. With the revised co-ordinates for the other atoms the value of R fell to 27·8%.

Two further rounds of Fourier and structure-factor calculations, with all atoms (except hydrogen) included in the structure-factor calculations, reduced the value of R to 24·7%. We then calculated F_o and F_c syntheses, derived back-shift corrections for termination-of-series errors and assigned individual isotropic temperature factors. Two atoms, C(24′) and C(28′), had rather lower peak heights in the F_o map than in the F_c map and were omitted from the next structure-factor calculation. The value of R was 22·7%.

A three-dimensional difference electron-density distribution calculated with Fourier coefficients ($F_o - F_c$) indicated slight shifts in atomic positions and when structure factors were recalculated with all atoms (except hydrogen) included the value of R was 22·0%. A second difference map was then calculated and further slight adjustments to the atomic co-ordinates and isotropic temperature factors were made. On calculation of a further set of structure factors the value of R was 21·4%.

The refinement of atoms away from pseudo-symmetric positions is a lengthy and somewhat unsatisfactory process.[13,14] In the present case we were unable to undertake a least-squares refinement, for the asymmetric unit comprises 66 atoms apart from hydrogen and the least-squares programme [24] for DEUCE is limited to a maximum of 64 atoms. Since, moreover, the study had reached a stage at which the chemical structure was clearly unambiguously established the analysis was concluded, although it was realized that the atomic co-ordinates were undoubtedly still approximate.

The theoretical atomic scattering factors derived by Berghuis *et al.*[25] for carbon and oxygen, by Tomiie and Stam [26] for chlorine, and by Freeman and Wood [27] for iron were used in all the structure-factor calculations; in the case of iron a correction for dispersion ($\Delta f'_K = -1{\cdot}72$) was applied.[28] The final calculated structure amplitudes and phase constants are listed with the measured values of the structure amplitudes in Table 4. The final three-dimensional electron-density distribution was evaluated on the basis of the phase constants of Table 4 and is shown in Fig. 2 by means of superimposed contour sections drawn parallel to (010). The final isotropic temperature factors are listed with the final atomic co-ordinates in Table 2.

The extensive calculations were performed on the Glasgow University DEUCE computer with programmes devised by Dr. J. S. Rollett and Dr. G. J. Sime. We thank the Carnegie Trust for a Scholarship (to I. C. P.).

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