The Crystal and Molecular
Structure of Leuco-thelephoric Acid Hexamethyl Ether

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A compound of unknown structure, extracted from the basidiomycete, *Corticium caeruleum*, has been shown to be the hexamethyl ether of leuco-thelephoric acid, $C_{24}H_{22}O_8$ (IUPAC name: 2,3,6,8,9,12-hexamethoxybenzobis-[1-2-*b*,4-5-*b'*]-benzofuran). The centrosymmetrical molecule has the outermost ring bent at $1\cdot3°$ from the coplanar central three rings but essentially shows considerable similarity to dibenzofuran. The average ring C–O bond length is $1\cdot390$ Å, and distortions of bond angles, reported previously for planar methoxybenzenoid compounds, are again present ($114\cdot5$ and $124\cdot5°$ for the two angles at the benzene ring). The crystals are monoclinic, $P2_1/c$, $a=7\cdot660$ (2), $b=8\cdot737$ (2), $c=15\cdot682$ (3) Å, $\beta=101\cdot41$ (2)°, $Z=2$, $D_x=1\cdot415$, $D_m=1\cdot41$ (1) g.cm^{-3}, Mo $K\alpha$ X-radiation, $\lambda=0\cdot7107$ Å). The structure was solved by direct methods using counter-measured X-ray data (maximum sin $\theta/\lambda=0\cdot605$, 1796 independent reflections, 305 unobserved). The R value is $0\cdot047$; heavy-atom bond lengths and angles have e.s.d.'s of $0\cdot003$ Å and *ca.* $0\cdot19°$ respectively.

Introduction

A very small quantity, literally a few crystals, of an unknown compound was isolated (Weisgraber, Weiss, Milne & Silverton, 1972) from the blue mycelium of the basidiomycete, *Corticium caeruleum*. The compound itself was colorless, ultraviolet spectroscopy indicated its aromatic nature, mass spectrometry gave a formula $C_{24}H_{22}O_8$, and n.m.r. spectroscopy confirmed the aromaticity and indicated that the molecule was probably symmetric and possessed six methoxyl groups. Because of the very small amount of the compound available, the X-ray study was undertaken with only the foregoing information.

Experimental

All programs used except those to be indicated were those of the X-ray 67 system of Stewart (1967). The basic experimental data are given in Table 1 and the

Table 1. *Crystal data*

Empirical formula	$C_{24}H_{22}O_8$
Formula weight	438·42
Systematic absences	$h0l$ for $l \neq 2n$
	$0k0$ for $k \neq 2n$
Space group	$P2_1/c$ (C_{2h}^5)
Radiation used for cell dimensions	Mo $K\alpha$, $\lambda=0\cdot7107$ Å
Temperature	*ca.* 23 °C
a	7·660 (2) Å
b	8·737 (2)
c	15·682 (3)
β	101·41 (2)°
V	1028·8 (4) Å^3
Z	2
$F(000)$	460
D_x	1·415 g cm^{-3}
D_m (Flotation in aqueous KI)	1·41 (1) g cm^{-3}
μ(Cu $K\alpha$) (data collection radiation)	9·04 cm^{-1}
Approximate dimensions of crystal used for data collection	0·30 × 0·25 × 0·22 mm

techniques used to obtain the data were standard ones as detailed in *e.g.* Beisler & Silverton (1971). Since there are only two molecules in the unit cell, the molecules must be centrosymmetric.

No clean single crystals were available and, although it was possible to remove attached surface material under a stereoscopic microscope, the crystal used for data collection had an irregular shape. The habit appeared to be monoclinic prismatic but the crystal had cross sections varying from irregular hexagonal to rounded-off rectangular. The crystal was mounted along the *b* axis for data collection and Cu $K\alpha$ X-radiation (Ni-filtered) was used. It is estimated that the average error in intensity caused by absorption was *ca.* 5%, an estimate essentially confirmed by measurement of $0k0$ reflections at different orientations. No absorption corrections were applied. The intensity data were collected using a Siemens automatic diffractometer. The moving-counter, moving-crystal method was used. A preliminary fast scan was employed followed by, if necessary, a repetition of the scan with attenuators inserted or else a slower scan to achieve, except for the weakest reflections, approximately constant counting statistics. A reflection was considered unobserved if the preliminary scan indicated that its integrated intensity, corrected for background, was less than three times its statistical standard deviation. The data were corrected for Lorentz and polarization factors and the 3σ value was inserted as the intensity for unobserved reflections. The control program allowed the use of only one standard reflection which was measured periodically without significant changes being observed. Further checks on stability were given by the measurement of about 100 reflections more than once and by the fact that diffracted intensities were measured, for two symmetrically related planes of the form $\{hk0\}$.

Structure determination and refinement

Quasinormalized structure-factor magnitudes, E values, were calculated by the *DATFIX* program of the X-ray 67 system. There are deviations in the distribution of E values from the centrosymmetric random atom model, but since the molecule was indicated as aromatic and hence probably planar, such deviations are not unexpected.

A sharpened, origin-removed, Patterson map proved

Table 2. *Observed and calculated structure factors* ($\times 10$)

The successive columns contain 1, F_o and F_1. Unobserved reflections are indicated by an asterisk and reflections omitted from the least-squares refinement by the letter E.

[Table 2 consists of an extensive multi-column listing of structure factor data (index, F_o, F_1) grouped by reflection classes; the individual numeric entries are not legibly reproducible.]

consistent with a planar aromatic compound having a molecular plane approximately parallel to ($\bar{2}$11) but was not immediately interpretable in terms of molecular structure.

Direct methods were employed to obtain the structure. A program of Bright & Cannon (1970, unpublished), *PHASE*1, was used to find and list $\sum_2$ relationships (Hauptman & Karle, 1953) for all E values greater than 1·4. The symbolic addition method (Karle & Karle, 1966) was then used both by hand and by means of a semi-automatic program *PHASE*11*A* written by the author. The criterion used in this study

for equating symbols was that such an apparent equality should occur in the symbolic phases of at least two different planes and, in each case, the lower of the appropriate values of $\sum\sigma_3\sigma_2^{-3/2}|E_hE_kE_{h-k}|$ had to exceed 3·3. Following Taga & Osaki (1971) the probability for equality of symbols would be at least 0·99999. It was not found difficult to achieve such a value but lower values could be used (probabilities of 0·999, 0·9995, 0·9999, 0·99995 are achieved by requiring lowest values of 2·07, 2·25, 2·65, 2·82 respectively).

The number of signs determined varied from 237 to 249 in four possible solutions. The only E map (Karle,

Table 3. *Atomic positions* ($\times 10^4$), *isotropic temperature factors before introduction of anisotropy and anisotropic temperature factors* ($\times 10^4$) *for heavier atoms.*

Estimated standard deriations are given parenthetically and refer to the last figure quoted. The anisotropic temperatuie factor used has the form: exp $[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$.

	x/a	y/b	z/c	B	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	5475 (3)	−42 (2)	940 (1)	3·2 (1)	150 (4)	116 (3)	25 (1)	−2 (3)	9 (1)	4 (1)
C(2)	5859 (3)	1215 (2)	462 (1)	3·1 (1)	133 (4)	105 (3)	29 (1)	−6 (3)	9 (1)	0 (1)
C(3)	6727 (3)	2666 (2)	707 (1)	2·9 (1)	131 (4)	92 (3)	30 (1)	0 (3)	13 (1)	2 (1)
C(4)	7539 (3)	3349 (2)	1497 (1)	3·1 (1)	164 (4)	99 (3)	29 (1)	7 (3)	13 (2)	4 (1)
C(5)	8278 (3)	4783 (2)	1472 (1)	3·2 (1)	161 (4)	100 (3)	31 (1)	1 (3)	12 (2)	−5 (1)
C(6)	8206 (3)	5556 (2)	−670 (1)	3·2 (1)	164 (4)	87 (3)	37 (1)	−5 (3)	17 (2)	−1 (1)
C(7)	7429 (3)	4889 (3)	−110 (1)	3·4 (1)	178 (4)	104 (3)	32 (1)	−9 (3)	13 (2)	8 (1)
C(8)	6710 (3)	3444 (2)	−61 (1)	3·0 (1)	152 (4)	105 (3)	29 (1)	−7 (3)	10 (1)	1 (1)
C(9)	5386 (3)	1232 (2)	−453 (1)	3·1 (1)	148 (4)	97 (3)	29 (1)	−10 (3)	13 (1)	8 (1)
C(10)	6463 (5)	−1316 (4)	2312 (2)	4·8 (1)	294 (7)	164 (4)	32 (1)	2 (5)	−4 (2)	21 (2)
C(11)	9103 (5)	4929 (4)	3015 (2)	5·0 (1)	297 (6)	169 (3)	32 (1)	−42 (4)	14 (2)	−5 (2)
C(12)	8942 (4)	7799 (3)	−42 (2)	4·2 (1)	253 (6)	100 (3)	45 (1)	−13 (4)	32 (2)	11 (2)
O(1)	5911 (2)	2598 (2)	−784 (1)	3·3 (1)	195 (3)	106 (2)	27 (1)	−29 (2)	8 (1)	5 (1)
O(2)	5901 (2)	52 (2)	1831 (1)	4·2 (1)	288 (4)	125 (2)	23 (1)	−15 (3)	7 (1)	3 (1)
O(3)	9138 (2)	5580 (2)	2187 (1)	4·1 (1)	251 (4)	106 (2)	31 (1)	−27 (2)	8 (1)	−5 (1)
O(4)	8974 (2)	6972 (2)	742 (1)	4·1 (1)	270 (4)	92 (2)	38 (1)	−36 (2)	23 (1)	1 (1)
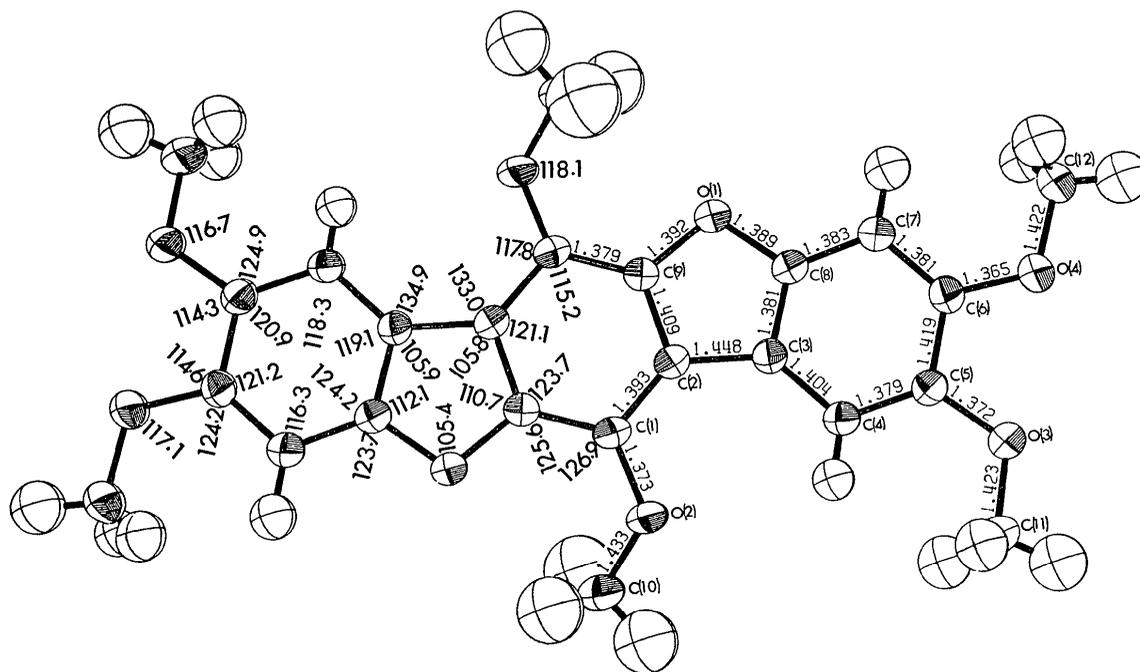
Fig. 1. Bond lengths and angles. The ellipsoids represent 40 % probability. E.s.d.'s in bond lengths and angles are ≤0·003 Å and ≤0·2° respectively.

Hauptman, Karle & Wing, 1958), leading to reasonable bond distances was that corresponding to the solution with the second highest number of sign determinations. This E map was also the only one without large variations in peak density.

Least-squares refinement minimizing $\sum w(F_o - F_c)^2$ was used. Unit weights were employed if $\Delta F < 7\cdot0$ (approximately three times the average standard deviation of F_o) and zero otherwise. In the last stages of refinement a ΔF cut-off of $4\cdot0$ was used. Unobserved reflections were included if F_c was greater than the 3σ threshold. Scattering factors were those for neutral carbon, nitrogen and oxygen given by International Tables for X-ray Crystallography (1962) and for bonded hydrogen by Stewart, Davidson & Simpson (1965). Least-squares refinement with isotropic temperature factors proceeded to an R value of $0\cdot132$ and confirmed that the compound was 2,3,6,8,9,12-hexamethoxybenzo-bis-[1-2-b, 4-5-b']-benzofuran, the hexamethyl ether of leuco-thelephoric acid. Further confirmation was given by the UV and IR spectra which are in good agreement with those published by Gripenberg (1960) who synthesized thelephoric acid and several of its derivatives.

A difference map, calculated after two cycles of refinement with anisotropic thermal parameters, had largest positive densities of $ca.$ $0\cdot2$ eÅ^{-3} except at possible hydrogen atom positions where the density varied from $0\cdot4$ to $0\cdot63$ eÅ^{-3}. Least-squares refinement with isotropic thermal parameters for hydrogen reduced the discrepancy to $0\cdot047$ (calculated for observed reflections only). In the final cycles calculated all shifts in heavier atom parameters were very small or were oscillating in sign. (The largest δ/σ was $0\cdot6$, the average value was $0\cdot24$ and $\sum[w(F_o - F_c)^2/[n-p])^{1/2}$ was $1\cdot51$.) In the final least-squares cycles, 10 planes had ΔF greater than $4\cdot0$ and were omitted. Eight of these planes had large intensities and the deviations may be caused by extinction since F_o is less than F_c in every case. The discrepancy for (080) was traced to a computer malfunction and the intensity measured for ($\bar{1}42$) will be modified by double reflection from (040) and ($\bar{1}02$), both of which have fairly large intensities. A list of observed and calculated structure factors is given as

Table 2, the atomic parameters are given in Tables 3 and 4.

Molecular geometry

Fig. 1, drawn by ORTEP (Johnson, 1965), shows the heavy-atom bond lengths and angles. Carbon–hydrogen bond lengths vary from $0\cdot96$ to $1\cdot06$ Å except for C(4)–H(1) which has a length of $0\cdot92$ Å. The e.s.d.'s of all hydrogen–carbon bond lengths are $0\cdot03$ Å except for those involving C(10) where the e.s.d.'s are $0\cdot04$ Å. The average C–H bond length is $0\cdot99$ Å and, given the e.s.d.'s, the deviations from the mean are not significant. The mean value is comparable with that observed in most X-ray studies of organic compounds. H–C–H angles range from 101 to 115° with a mean of $109\cdot5°$ and an average e.s.d. of $2\cdot7°$.

The molecule is essentially flat as may be seen from Fig. 2 which presents the deviations from various least-squares planes. There are small deviations from planarity of the five linked rings but deviations in the individual rings are not significant. Referred to axes along a^*, b, c, the mean plane for all five rings has the equation: $0\cdot9108x - 0\cdot4077y - 0\cdot0654z - 3\cdot4883 = 0$ and the planes of rings 1, 2 and 3 have equations: $0\cdot9125x - 0\cdot4053y - 0\cdot0557z - 3\cdot4948 = 0$, $0\cdot9125x - 0\cdot4054y - 0\cdot0544z - 3\cdot4958 = 0$ and $0\cdot9077x - 0\cdot4129y - 0\cdot0750z - 3\cdot4402 = 0$ respectively. Rings 1 and 2 are essentially coplanar and ring 3 makes an angle of $1\cdot3°$ with the plane of the central three rings.

The average bond length in the central benzene ring is $1\cdot394$ Å and that in the outside ring is $1\cdot391$ Å. The difference is not significant and neither of these values differs significantly from that quoted by Sutton, Kennard, Powell & Whiffen (1965). It is possible that the apparent shortening of the average bond length of the outer ring is a consequence of molecular libration, but no corrections have been applied. The individual bond lengths in the rings differ significantly from each other and from the means but variation is not unexpected since all but four carbon atoms of the ring system are involved in ring fusion or have methoxyl substituents.

Table 4. Positional parameters ($\times 10^4$) and isotropic temperature factors for hydrogen atoms

Estimated standard deviations are given parenthetically and refer to the last figure quoted.

	x/a	y/b	z/c	B
H(1)	762 (3)	288 (3)	203 (1)	3·8 (5)
H(2)	733 (3)	544 (3)	−69 (2)	4·8 (6)
H(3)	538 (5)	813 (4)	237 (2)	10·0 (1·1)
H(4)	725 (5)	807 (5)	198 (3)	12·6 (1·2)
H(5)	715 (5)	901 (4)	287 (2)	9·4 (1·0)
H(6)	788 (4)	492 (3)	311 (2)	5·8 (7)
H(7)	973 (4)	565 (3)	343 (2)	7·0 (8)
H(8)	964 (4)	381 (3)	306 (2)	6·4 (7)
H(9)	946 (3)	728 (3)	−47 (1)	4·9 (6)
H(10)	772 (4)	795 (3)	63 (2)	6·3 (7)
H(11)	954 (4)	879 (3)	11 (2)	6·6 (8)

Fig. 2. Deviations of atoms from mean planes (Å × 10⁴). The figures on the right of the drawing represent deviations from the mean plane of all five rings and those to the left (italicized) represent deviations from the individual ring mean planes.

A recent paper by Dideberg, Dupont & André (1972) discusses the structure of dibenzofuran and essentially completes the study of simple dibenzo hetero-pentacyclic compounds. In the present study the required molecular symmetry is $\bar{1}$, while in dibenzofuran the symmetry is m. Corresponding bond lengths and angles are, however, very similar (Table 5). As in dibenzofuran, individual rings in leuco-thelephoric acid hexamethyl ether are planar but, as opposed to dibenzofuran where each benzene ring makes an angle of 1·2° with the furan ring, the central three rings in the present study are essentially planar and the outermost benzene rings are at angles of 1·3° to the central ring system. Some other differences may be attributable to the larger extended conjugated system. The C–O bond length in the furan ring is shorter than that in dibenzofuran (the difference, 0·013 Å, is ca. 3σ). However, in neither compound is there a very strong indication of conjugation since, although the C–O bond length in the present study is 0·036 Å shorter than that quoted by Sutton et al. (1965) for straight chain compounds, this difference is comparable with that between an sp^3–sp^3 and an sp^2–sp^3 C–C bond (0·027 Å).

Table 5. Comparison of furan ring dimensions in dibenzofuran, leuco-thelephoric acid hexamethyl ether and furan

	Dibenzo-furan	Leuco-thele-phoric acid	Furan*
C–O	1·404 Å	1.392, 1·389 Å	1·362 Å
C–C (benzene)	1·393	1·409, 1·381	1·361
C–C (furan)	1·481	1·448	1·431
C–O–C	104·1°	105·4°	106·55°
C–C–O	112·3	110·7° 112·1°	110·68
C–C–C	105·3	105·8 105·9	106·06

* r_s values from Bak, Christensen, Dixon, Hansen–Nygaard, Rastrup–Andersen & Schottlaender (1962).

The formally single C–C bond in the furan ring of leuco-thelephoric acid hexamethyl ether is somewhat shorter than that in dibenzofuran and possibly the more extended conjugation systemis again responsible. The actual length, 1·448 Å, is more directly comparable with dibenzothiophen (1·441 Å) and dibenzoselenophen (1·453 Å), as listed by Dideberg, Dupont & André (1972), and also with furan (1·431 Å: Bak, Christensen, Dixon, Hansen-Nygaard, Rastrup-Anderson & Schottlaender,

1962) and thiophen (1·423 Å: Bak, Christensen, Hansen-Nygaard & Rastrup-Andersen, 1961).

The averages of the bond lengths C (aromatic)–O(methoxyl): 1·370 Å and O–C(methyl): 1·426 Å, are consistent with those given by Sutton et al. (1965). The average C(aromatic)–O–C(methyl) angle is 117·3° and this value is again quite normal.

The enlargement of angles C(4)–C(5)–O(3) and C(7)–C(6)–O(4) to 124·9 and 124·2° respectively is similar to that observed in planar methoxy benzenoid compounds, e.g. deoxyanisoin and p,p-dimethoxybenzophenone (Norment & Karle, 1962) and anisic acid (Bryan, 1967) and can be attributed, following Norment and Karle, to close contacts between benzenoid and methyl hydrogen atoms. It is interesting that the values in this o-dimethoxy compound are quite comparable with those in monomethoxy compounds and that repulsion between the oxygen atoms seems less important than H–H repulsion [perhaps packing forces may reinforce the repulsion since the shortest intermolecular approaches involve O(3) and O(4)].

The inequality of the corresponding angles at C(1), viz. 126·9 and 117·8°, cannot be attributed to the same causes nor even to lone-pair repulsion since the lone pairs of electrons on the ring oxygen atom and the methoxyl oxygen atom point in quite different directions. Possibly packing forces are involved since C(10) is fairly close (3·428 Å) to O(3) in the molecule related by the b axis.

Molecular packing

A stereo diagram showing the molecular packing, produced by ORTEP, is given as Fig. 3. There are similarities to the packing seen in linear condensed ring hydrocarbons, e.g. naphthalene (Abrahams, Robertson & White, 1969) and anthracene (Sinclair, Robertson & Mathieson, 1950) in that a similar 'herringbone' packing is adopted but the direction of molecular elongation is more nearly along a + b instead of along a. Thus, although the angle of inclination of the molecular plane to the (010) plane is 65·9° and thus similar to the angle in naphthalene and anthracene, there are considerable differences from the cell dimensions observed for the hydrocarbons, aside from obvious differences expected from the larger methoxyl-substituted molecule.
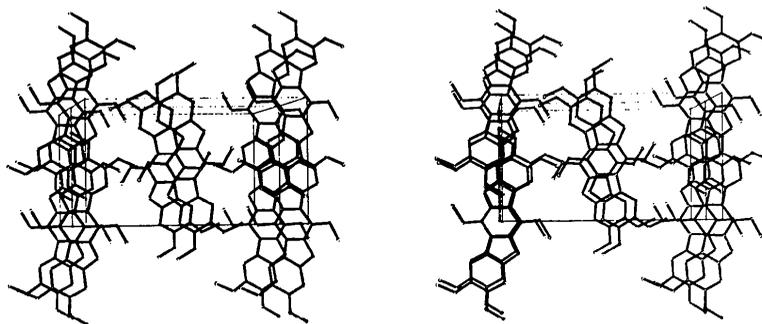
Fig. 3. Stereoscopic drawing of two unit cells to show packing. The direction of projection is along a.*

Apart from C(10), all heavy atoms and one hydrogen atom from each of those attached to C(10), C(11)–C(12) are essentially coplanar. It can be seen from Fig. 3 that C(10) can twist out of the molecular plane into a gap in the crystal. The weaker forces holding the C(10) methyl group seem reflected in the high thermal parameters of its hydrogen atoms H(3), H(4), H(5). No heavy atom intermolecular contacts are excessively short, all being longer than 3·5 Å except for C(10)–O(3): 3·428 (4) Å (related by **b**), C(8)–C(12): 3·477 (4) Å related by **a**+**b** and Ī) and O(4)–C(11): 3·391 (2) Å (related by 2_1).

The large E values of the orders of ($\bar{2}$11) are readily explained by the fact that the mean plane of the rings makes an angle of 1·3° with ($\bar{2}$11) and that all heavier atoms are within 0·1 Å of the plane except for O(3) and C(10) which are 0·18 and 0·68 Å distant respectively.

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Structures Cristallines et Moléculaires de Trois Formes Polymorphes de l'Oestrone

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The crystal and molecular structures of three polymorphous forms of estrone have been determined by X-ray diffraction. Two forms (estrone I and II) crystallize as orthorhombic crystals, $P2_12_12_1$, $Z=4$, $a=12·88$, $b=16·301$, $c=7·463$ and $a=10·043$, $b=18·424$, $c=7·787$ Å and the third as a monoclinic crystal, $P2_1$, $Z=4$ (2 independent molecules) $a=9·271$, $b=22·285$, $c=7·610$ Å, $\beta=111·45°$. The three structures have been solved by direct methods and refined by least-squares computations to R values of 0·069 for estrone I, 0·039 for estrone II and 0·061 for estrone III. The conformation of the four molecules shows the flexibility of the B and D rings of the steroid. Nonetheless it is impossible, with a calculated model, to report an accurate conformation of these parts of the molecule. The crystalline cohesions are different in the three structures. Estrones I and III have layers of parallel molecules linked by hydrogen bonds and estrone II has a herringbone arrangement with a weaker hydrogen bond and stronger dispersion bonds.

Dans le cadre de l'étude des hormones stéroides oestrogènes que nous avons entreprise (Busetta & Hospital, 1969, 1972; Busetta, Courseille, Geoffre & Hospital, 1972), nous présentons ici les structures des trois formes polymorphes de l'oestrone. L'oestrone ($C_{18}H_{22}O_2$) (Fig. 1) est un stéroide qui a longtemps été considéré