Table 4. *Interatomic distances* (Å) *and angles* (°) *in the hydrogen bonds*

a *b* *c*	*ab*	*bc*	*ac*	∠*abc*
N(1′)–H···O(1) $(-x+1, -y+1, -z)$	0·792 (2)	2·108 (2)	2·854 (3)	157·3 (2)
N(3′)–H···N(6) $(x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2})$	0·981 (2)	1·974 (2)	2·930 (3)	164·4 (2)

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References

DUAX, W. L. & NORTON, D. A. (1975). *Atlas of Steroid Structure.* New York: Plenum.

FLORENCIO, F., SMITH-VERDIER, P. & GARCÍA-BLANCO, S. (1978*a*). *Acta Cryst.* B**34**, 1317–1321.

FLORENCIO, F., SMITH-VERDIER, P. & GARCÍA-BLANCO, S. (1978*b*). *Acta Cryst.* B**34**, 2220–2223.

GONZALEZ-TRIGO, J., AVENDAÑO, C. & MARTINEZ-MORENO, M. (1974). *Pharm. Mediterr.* **10**, 639–653.

HENDRICKSON, J. B. (1961). *J. Am. Chem. Soc.* **83**, 4537–4547.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 72–98. Birmingham: Kynoch Press.

KLYNE, W. & PRELOG, V. (1960). *Experientia*, **16**, 521–523.

MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1974). *MULTAN 74. A System of Computer Programs for the Automatic Solution of Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

SMITH-VERDIER, P., FLORENCIO, F. & GARCÍA-BLANCO, S. (1977). *Acta Cryst.* B**33**, 3381–3385.

SMITH-VERDIER, P., FLORENCIO, F. & GARCÍA-BLANCO, S. (1979). *Acta Cryst.* B**35**, 216–217.

Acta Cryst. (1979). B35, 2424–2427

Structure of 3a,5-Diethyl-4-oxo-6,6a-diphenyl-3-(2,4,6-trimethylphenyl)-3a,4-dihydrocyclopenta[2,3-*d*]isoxazoline (TPDCI)

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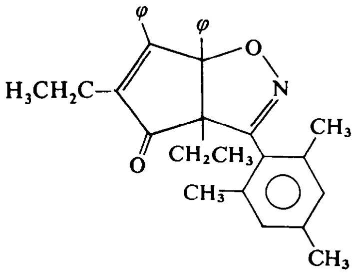
Abstract. $C_{31}H_{31}NO_2$, M_r = 449·566, monoclinic, $P2_1/n$, a = 19·440 (2), b = 14·681 (1), c = 8·900 (1) Å, β = 95·02 (1)°, Z = 4, V = 2530·16 Å^3, λ(Mo $K\alpha$) = 0·71069 Å, F(000) = 960, D_c = 1·178, D_m = 1·164 Mg m^{-3}, m.p. 468–469 K, μ = 0·078 mm^{-1}; 1223 independent non-zero reflexions. The structure was solved by direct phase determination with *MULTAN*. The positional and vibrational parameters, with anisotropic temperature factors for the non-hydrogen atoms, were refined by full-matrix least-squares calculations to a final R = 0·068. The molecule of the compound has a cyclopentenone–isoxazoline structure. To the two central five-membered isoxazoline and cyclopentenone rings, at an angle of 113·20°, are linked three benzene rings and two ethyl groups with different orientations. Unlike other isoxazoline derivatives, the isoxazoline ring in this case is almost perpendicular to the trimethyl-substituted benzene ring, instead of forming a roughly coplanar system with it.

Introduction. In two previous papers (Rodiou, Kokkou & Rentzeperis, 1978; Stergiou, Kokkou & Rentzeperis, 1978) the structures of two new isoxazoline derivatives, prepared at the Laboratory of Organic Chemistry of the Aristotle University of Thessaloniki (Alexandrou & Argyropoulos, 1977), were investigated. The present structure determination of TPDCI is the third of a series under investigation.



Pure, colourless TPDCI crystals were kindly provided by Professor N. E. Alexandrou and Dr N. Argyropoulos. A transparent prismatic single crystal, with dimensions 0·25 × 0·27 × 0·29 mm, was selected and centred on our computer-controlled Philips PW 1100 four-circle single-crystal diffractometer. With Mo $K\alpha$ radiation (λ = 0·71069 Å) the cell constants were determined by measuring the θ angles of 200 strong reflexions with large θ values directly on the diffractometer and subsequently processing them with the least-squares program *PARAM* [part of the XRAY system of crystallographic programs (Stewart, Kruger, Ammon, Dickinson & Hall, 1976)]. Systematic absences led to the space group $P2_1/n$. The density of the crystals was measured by flotation in a potassium bromide solution.

Three-dimensional intensity data were collected with a scintillation counter on the PW 1100 diffractometer in the ω-scan mode, using Mo $K\alpha$ radiation and a graphite monochromator. The intensities of 3642 independent reflexions up to 2θ = 47·2° (max. hkl = 20,15,±10 respectively) were examined and measured. Of these, 1223 with intensities greater than 2σ were considered as observed and included in all subsequent computations.

Table 1. *Atomic coordinates* (×10⁴) *and isotropic temperature factors for the non-hydrogen atoms in TPDCI*

Here and throughout this paper the e.s.d.'s are given in parentheses and refer to the last digit.

	x	y	z	B (Å^2)
O(1)	2247 (3)	8288 (5)	1999 (7)	3·4
O(2)	466 (3)	6671 (5)	3217 (7)	4·4
N	1921 (4)	7858 (6)	700 (8)	3·3
C(1)	1286 (5)	7708 (7)	894 (11)	3·0
C(2)	1054 (4)	8029 (6)	2413 (10)	2·6
C(3)	952 (5)	7191 (7)	3402 (11)	3·2
C(4)	1508 (5)	7132 (7)	4614 (11)	3·0
C(5)	1957 (5)	7809 (7)	4487 (10)	2·6
C(6)	1751 (4)	8429 (7)	3149 (10)	2·7
C(7)	822 (5)	7258 (8)	9689 (12)	3·0
C(8)	409 (6)	7761 (7)	8630 (14)	4·0
C(9)	9973 (6)	7285 (10)	7592 (13)	4·8
C(10)	9909 (5)	6354 (9)	7558 (12)	4·0
C(11)	360 (6)	5884 (7)	8549 (13)	3·9
C(12)	817 (5)	6292 (7)	9622 (11)	2·9
C(13)	397 (5)	8606 (7)	2292 (11)	3·7
C(14)	101 (5)	8797 (8)	3818 (13)	5·0
C(15)	1523 (6)	6357 (8)	5744 (11)	4·6
C(16)	2102 (7)	5680 (9)	5646 (16)	8·0
C(17)	2597 (4)	7996 (6)	5481 (10)	2·8
C(18)	3240 (5)	8133 (7)	4878 (10)	3·5
C(19)	3823 (5)	8259 (8)	5842 (13)	4·7
C(20)	3790 (4)	8280 (8)	7391 (12)	4·3
C(21)	3160 (5)	8167 (8)	7979 (10)	4·2
C(22)	2578 (5)	8009 (7)	7035 (11)	3·9
C(23)	1741 (5)	9440 (7)	3471 (12)	3·5
C(24)	1721 (5)	9798 (8)	4889 (13)	4·0
C(25)	1701 (6)	731 (10)	5126 (14)	5·1
C(26)	1707 (6)	1304 (9)	3895 (19)	6·0
C(27)	1725 (7)	987 (9)	2484 (18)	6·7
C(28)	1749 (6)	43 (8)	2250 (13)	4·9
C(29)	469 (6)	8781 (8)	8510 (13)	5·6
C(30)	9413 (6)	5869 (9)	6420 (12)	6·5
C(31)	1310 (6)	5726 (8)	646 (11)	4·9

Table 2. *Atomic coordinates* (×10³) *and isotropic temperature factors for the hydrogen atoms in TPDCI*

	x	y	z	B (Å^2)
H(C9)	958 (3)	757 (4)	685 (7)	1·8 (1·6)
H(C11)	35 (3)	528 (4)	862 (7)	1·5 (1·4)
H(C18)	324 (4)	815 (6)	367 (8)	4·3 (2·7)
H(C19)	428 (4)	839 (6)	541 (9)	5·5 (2·9)
H(C20)	429 (4)	851 (5)	798 (8)	3·6 (2·4)
H(C21)	311 (3)	813 (5)	915 (7)	2·2 (2·0)
H(C22)	216 (5)	786 (7)	744 (11)	7·7 (3·6)
H(C24)	172 (3)	932 (5)	580 (7)	1·6 (1·5)
H(C25)	171 (6)	100 (8)	605 (13)	9·1 (4·8)
H(C26)	183 (6)	192 (9)	415 (13)	12·0 (5·2)
H(C27)	161 (8)	137 (12)	168 (19)	17·7 (7·6)
H(C28)	174 (4)	978 (5)	117 (8)	2·7 (2·5)
H1(C13)	49 (5)	918 (6)	199 (10)	5·7 (3·2)
H2(C13)	8 (3)	824 (4)	152 (6)	1·8 (1·6)
H1(C14)	968 (5)	926 (6)	356 (10)	5·6 (3·3)
H2(C14)	987 (5)	825 (7)	425 (10)	9·7 (3·3)
H3(C14)	48 (4)	917 (5)	447 (8)	3·1 (2·7)
H1(C15)	164 (4)	658 (6)	677 (8)	3·2 (2·5)
H2(C15)	104 (6)	598 (8)	554 (13)	10·7 (4·7)
H1(C16)	205 (6)	544 (9)	477 (14)	14·5 (4·6)
H2(C16)	201 (6)	524 (10)	640 (15)	13·2 (5·5)
H3(C16)	262 (6)	608 (8)	597 (13)	9·4 (4·4)
H1(C29)	16 (7)	911 (8)	912 (14)	7·3 (5·0)
H2(C29)	36 (6)	895 (7)	743 (13)	7·3 (4·1)
H3(C29)	98 (6)	903 (7)	899 (12)	9·4 (4·2)
H1(C30)	953 (7)	575 (9)	550 (16)	8·8 (5·6)
H2(C30)	894 (8)	610 (10)	634 (17)	15·0 (6·7)
H3(C30)	953 (8)	524 (11)	652 (18)	18·6 (6·7)
H1(C31)	157 (8)	597 (10)	147 (17)	13·4 (6·3)
H2(C31)	148 (5)	530 (8)	6 (13)	8·7 (4·1)
H3(C31)	115 (6)	521 (10)	142 (15)	10·9 (5·2)

Integrated intensities were converted to $|F_o|$ values in the usual way, using the special measurement treatment program *DATRED* (Main, 1970). Since μ is very small, no absorption correction was applied. For further calculations the programs of the *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974) and XRAY systems were used.

The structure was essentially solved by direct phase determination with the *MULTAN* system. The phases of 280 strong reflexions were determined and on the resulting E map it was possible to locate all the 34 non-hydrogen atoms of the asymmetric unit. A structure factor calculation at this stage, with the 280 reflexions and an overall B = 1·10 Å^2, gave R = 0·285.

Refinement of the structure was carried out by full-matrix least-squares calculations with the XRAY system. The atomic scattering factors for O, N and C were taken from Cromer & Waber (1965), and for H from Stewart, Davidson & Simpson (1965). A single scale factor was used for the whole set of reflexion data.

With the same overall temperature factor and unit weights, refinements reduced R to 0·161 in six cycles.

Table 3. *Interatomic distances* (Å) *in TPDCI*

O(1)–N	1·42 (1)	C(12)–C(7)	1·42 (2)
N–C(1)	1·28 (1)	C(8)–C(29)	1·51 (2)
C(1)–C(2)	1·54 (1)	C(10)–C(30)	1·51 (2)
C(2)–C(6)	1·57 (1)	C(12)–C(31)	1·51 (1)
C(6)–O(1)	1·48 (1)	C(13)–C(14)	1·55 (2)
C(1)–C(7)	1·49 (1)	C(15)–C(16)	1·51 (2)
C(2)–C(13)	1·53 (1)		
C(6)–C(23)	1·51 (1)	C(17)–C(18)	1·42 (1)
		C(18)–C(19)	1·37 (1)
C(2)–C(3)	1·54 (1)	C(19)–C(20)	1·39 (2)
C(3)–C(4)	1·46 (1)	C(20)–C(21)	1·38 (1)
C(4)–C(5)	1·33 (1)	C(21)–C(22)	1·37 (1)
C(5)–C(6)	1·52 (1)	C(22)–C(17)	1·39 (1)
C(3)–O(2)	1·21 (1)		
C(4)–C(15)	1·52 (1)	C(23)–C(24)	1·37 (2)
C(5)–C(17)	1·49 (1)	C(24)–C(25)	1·39 (2)
		C(25)–C(26)	1·38 (2)
C(7)–C(8)	1·39 (2)	C(26)–C(27)	1·34 (2)
C(8)–C(9)	1·39 (2)	C(27)–C(28)	1·40 (2)
C(9)–C(10)	1·37 (2)	C(28)–C(23)	1·40 (2)
C(10)–C(11)	1·37 (2)		
C(11)–C(12)	1·38 (1)		

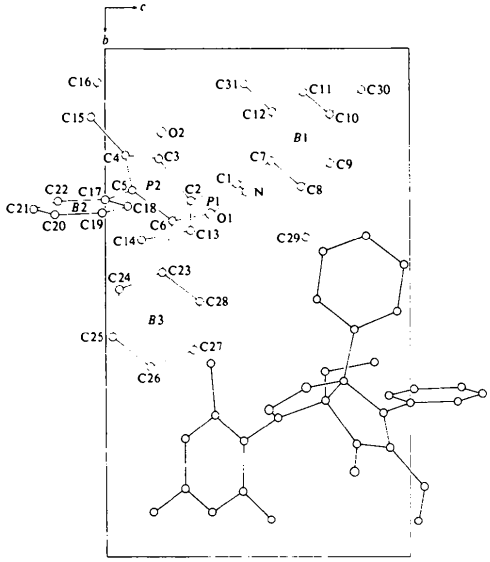


Fig. 1. Projection of a TPDCI molecule parallel to (010).

Table 4. *Bond angles* (°) *in TPDCI*

C(6)–O(1)–N	110·8 (0·6)	C(7)–C(8)–C(29)	122·1 (1·0)
O(1)–N–C(1)	109·5 (0·7)	C(29)–C(8)–C(9)	120·0 (1·0)
N–C(1)–C(2)	115·2 (0·8)	C(8)–C(9)–C(10)	124·4 (1·1)
N–C(1)–C(7)	120·6 (0·9)	C(9)–C(10)–C(11)	115·8 (1·0)
C(2)–C(1)–C(7)	124·3 (0·9)	C(9)–C(10)–C(30)	122·3 (1·0)
C(1)–C(2)–C(6)	100·0 (0·7)	C(30)–C(10)–C(11)	121·7 (1·1)
C(1)–C(2)–C(13)	114·5 (0·7)	C(10)–C(11)–C(12)	124·1 (1·0)
C(1)–C(2)–C(3)	108·8 (0·8)	C(11)–C(12)–C(7)	117·6 (0·9)
C(13)–C(2)–C(6)	120·5 (0·8)	C(11)–C(12)–C(31)	120·9 (0·9)
C(2)–C(6)–C(23)	115·0 (0·7)	C(31)–C(12)–C(7)	121·5 (0·9)
C(2)–C(6)–O(1)	104·5 (0·7)		
C(5)–C(6)–O(1)	108·3 (0·7)	C(2)–C(13)–C(14)	114·3 (0·8)
C(23)–C(6)–O(1)	106·7 (0·8)		
		C(4)–C(15)–C(16)	115·2 (1·0)
C(3)–C(2)–C(6)	102·2 (0·7)		
C(3)–C(2)–C(13)	109·6 (0·8)	C(5)–C(17)–C(18)	121·4 (0·8)
C(2)–C(3)–C(4)	110·3 (0·8)	C(5)–C(17)–C(22)	120·1 (0·8)
C(2)–C(3)–O(2)	124·3 (0·8)	C(18)–C(17)–C(22)	118·4 (0·8)
O(2)–C(3)–C(4)	125·4 (0·9)	C(17)–C(18)–C(19)	119·3 (0·9)
C(3)–C(4)–C(5)	109·9 (0·9)	C(18)–C(19)–C(20)	121·2 (0·9)
C(3)–C(4)–C(15)	120·3 (0·9)	C(19)–C(20)–C(21)	119·5 (0·8)
C(15)–C(4)–C(5)	129·8 (0·8)	C(20)–C(21)–C(22)	120·0 (0·9)
C(4)–C(5)–C(17)	127·3 (0·8)	C(21)–C(22)–C(17)	121·5 (0·9)
C(4)–C(5)–C(6)	112·5 (0·8)		
C(17)–C(5)–C(6)	120·2 (0·8)	C(6)–C(23)–C(24)	123·5 (1·0)
C(5)–C(6)–C(2)	105·1 (0·7)	C(6)–C(23)–C(28)	118·2 (0·9)
C(5)–C(6)–C(23)	116·4 (0·8)	C(24)–C(23)–C(28)	118·3 (1·0)
		C(23)–C(24)–C(25)	121·5 (1·1)
C(1)–C(7)–C(8)	121·7 (1·0)	C(24)–C(25)–C(26)	118·5 (1·2)
C(1)–C(7)–C(12)	118·3 (0·9)	C(25)–C(26)–C(27)	122·3 (1·2)
C(8)–C(7)–C(12)	120·0 (0·9)	C(26)–C(27)–C(28)	119·0 (1·3)
C(7)–C(8)–C(9)	117·7 (1·0)	C(27)–C(28)–C(23)	120·4 (1·1)

Five further cycles with individual isotropic temperature factors lowered R to 0·140. The three subsequent cycles with anisotropic temperature coefficients gave R = 0·101. The H atoms were then located by using the program *BONDAT* of the XRAY system and included in six further refinement cycles with isotropic temperature factors assigned to them (equal to those of the corresponding C atoms to which they are bonded); R reduced to 0·069. At this stage a systematic weight analysis was carried out which led to the following weighting scheme: $w = 1/\{1 + [(F - B)/A]^2\}$ with $A = 21{\cdot}5$, $B = 50{\cdot}0$. However, the value of R remained almost the same in the two subsequent cycles (R = 0·068, R_w = 0·063). The average shift/error ratio of the last cycle is less than 0·26.

The final positional parameters and isotropic temperature coefficients for the non-hydrogen atoms are given in Table 1.* The final coordinates and isotropic temperature factors for the H atoms are shown in Table 2. Interatomic distances and bond angles are given in Tables 3 and 4.

Discussion. The structural and conformational features of the TPDCI molecule are shown in the projection of Fig. 1. The compound is a cyclopentenone–isoxazoline derivative.

The two central five-membered isoxazoline (*P*1) and cyclopentenone (*P*2) rings form an angle of 113·2° with each other, which compares very well with the 107° found in the case of 3-(*p*-chlorophenyl)-4-oxo-3a,5,6,6a-tetraphenyl-3a,4-dihydrocyclopenta[2,3-*d*]-isoxazoline (CPCI in the following; Rodiou, Kokkou & Rentzeperis, 1978), and the 110° found in 3a,5-bis(methoxycarbonyl)-3-(*p*-nitrophenyl)-4-oxo-6,6a-

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* Lists of structure factors, anisotropic thermal parameters, bond distances and angles involving H atoms and least-squares planes through the benzene rings have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34517 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 5. *Least-squares planes in TPDCI, with displacements of atoms from the planes* (Å)

The equation for a plane is in the form $AX + BY + CZ = D$ and refers to an orthogonal system of axes, with $X \parallel \mathbf{a}$, $Y \parallel \mathbf{b}$, $Z \parallel \mathbf{c}^*$ and D, the distance of the plane from the origin, in Å. Asterisks indicate atoms not included in the calculation of the plane. Mean estimated standard deviations of the atoms defining a plane are given in parentheses attached to the distance of the first atom.

Plane $P1$: five-membered isoxazoline ring

$$-0{\cdot}20801X + 0{\cdot}89407Y - 0{\cdot}39669Z = 9{\cdot}30027$$

O(1)	−0·001 (2)	C(2)	0·002
N	0·002	C(6)	−0·001
C(1)	−0·003	C(7)*	−0·002

Plane $P2$: five-membered cyclopentenone ring

$$0{\cdot}57216X - 0{\cdot}56967Y - 0{\cdot}59001Z = -6{\cdot}89885$$

C(2)	−0·01 (1)	C(6)	0·01
C(3)	0·01	O(2)*	0·01
C(4)	−0·01	C(15)*	0·02
C(5)	−0·00	C(17)*	−0·01

Dihedral angles between planes (°) [$B1$, $B2$, $B3$ are the benzene rings (see Fig. 1)]

$B1 \wedge B2$ 83·9	$B2 \wedge B3$ 81·9	$B3 \wedge P1$ 76·7	$P1 \wedge P2$ 66·8
$B1 \wedge B3$ 46·5	$B2 \wedge P1$ 21·4	$B3 \wedge P2$ 60·7	
$B1 \wedge P1$ 85·0	$B2 \wedge P2$ 51·0		
$B1 \wedge P2$ 34·6			

diphenyl-3a,4-dihydrocyclopenta[2,3-*d*]isoxazoline (NPCCI in the following; Stergiou, Kokkou & Rentzeperis, 1978). To these rings are linked three benzene rings and two ethyl groups ($E1$, $E2$) with different orientations. The benzene rings $B1$, $B2$, $B3$ and the five-membered $P1$ and $P2$ (Fig. 1) are planar to a good approximation. In Table 5 are listed the least-squares planes through $P1$ and $P2$, together with the dihedral angles between them and the planes of the benzene rings.

Contrary to CPCI and NPCCI mentioned above, where the characteristic feature of the molecule is the coplanar system formed by the isoxazoline ring $P1$ and the benzene ring $B1$, in this case $B1$ is almost perpendicular to $P1$ ($B1 \wedge P1$ = 85°), evidently due to the presence of the three methyl groups substituted in $B1$.

The geometrical features of the benzene rings are in good agreement with the usually accepted values. Mean bond lengths of the rings $B1$, $B2$ and $B3$ are 1·39 (2), 1·39 (1) and 1·38 (2) Å respectively.

The general features of the five-membered isoxazoline and cyclopentenone rings are normal. The two ethyl groups, $E1$ [connected to C(2)] and $E2$ [connected to C(4)], show normal bond lengths.

The intramolecular contact distances O(2)···H2(C15) = 2·48 (11) Å and O(1)···H(C28) = 2·49 (8) Å almost coincide with the sum (2·48 Å) of the van der Waals radii of the corresponding atoms (Bondi, 1964); the distance O(1)···H(C18) = 2·34 (7) Å does not deviate much from this value. All other contact distances are within the expected values.

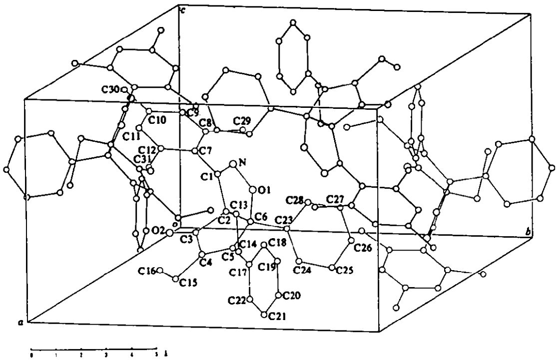

Fig. 2. Clinographic projection of the unit cell, showing the molecular packing.

Fig. 2 is a clinographic projection of the structure showing the molecular packing of TPDCI in the unit cell. The various intermolecular distances are normal.

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References

ALEXANDROU, N. E. & ARGYROPOULOS, N. (1977). 6th International Congress of Heterocyclic Chemistry, Teheran.

BONDI, A. (1964). *J. Phys. Chem.* **68**, 441–451.

CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.

MAIN, P. (1970). Private communication. Univ. of York, England.

MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1974). *MULTAN, A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain-la-Neuve, Belgium.

RODIOU, D. C., KOKKOU, S. C. & RENTZEPERIS, P. J. (1978). *Acta Cryst.* B**34**, 562–567.

STERGIOU, A. C., KOKKOU, S. C. & RENTZEPERIS, P. J. (1978). *Acta Cryst.* B**34**, 1662–1666.

STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. & HALL, S. R. (1976). The XRAY system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.