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### References

- AHMED, F. R., HALL, S. R., PIPPY, M. E. & HUBER, C. P. (1973). NRC Crystallographic Programs for the IBM/360 System. Accession Nos. 133–147 in *J. Appl. Cryst.* (1973), **6**, 309–346.
- ARNOTT, S., DAVIE, A. W., ROBERTSON, J. M., SIM, G. A. & WATSON, D. G. (1961). *J. Chem. Soc.* pp. 4183–4200.
- ÁSBRINK, J. & WERNER, P. E. (1966). *Acta Cryst.* **20**, 407–410.
- BERKING, B. & SEEMAN, N. C. (1971). *Acta Cryst.* **B27**, 1752–1760.
- BIRNBAUM, G. I. (1973). *Acta Cryst.* **B29**, 1426–1432.
- BLANK, F., BUXTORF, C., CHIN, O., JUST, G. & TREDOR, J. G. (1969). *Can. J. Chem.* **47**, 1561–1570.
- BRISSE, F., LECTARD, A. & SCHMIDT, C. (1974). *Can. J. Chem.* **52**, 1123–1134.
- COPPENS, P. & HAMILTON, W. C. (1970). *Acta Cryst.* **A26**, 71–83.
- CRAVEN, B. M. (1962). *Acta Cryst.* **15**, 387–396.
- CRAVEN, B. M. (1964). *Acta Cryst.* **17**, 396–403.
- CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.
- CUNNINGHAM, G. L., BOYD, A. W., MYERS, R. J., GWINN, W. D. & LEVAN, W. J. (1951). *J. Chem. Phys.* **19**, 676–685.
- DUSAUSOY, Y., PROTAS, J., BESANCON, J. & TIROUFLET, J. (1973). *Acta Cryst.* **B29**, 469–476.
- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1970). *Acta Cryst.* **B26**, 274–285.
- GRANT, I. G., HAMILTON, J. A., HAMOR, T. A., ROBERTSON, J. M. & SIM, G. A. (1963). *J. Chem. Soc.* pp. 2506–2515.
- MACKAY, M. F. & MATHIESON, A. M. L. (1965). *Acta Cryst.* **19**, 417–425.
- MATHEWS, D. A., SWANSON, J., MUELLER, M. H. & STUCKY, G. D. (1971). *J. Am. Chem. Soc.* **93**, 5945–5953.
- MERLINO, S., LAMI, G., MACCHIA, B., MACCHIA, F. & MONTI, L. (1972). *J. Org. Chem.* **37**, 703–706.
- NG, A. S., JUST, G. & BLANK, F. (1969). *Can. J. Chem.* **47**, 1223–1227.
- RICHE, C. (1973). *Acta Cryst.* **B29**, 2154–2160.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- SUTTON, L. E. (1965). *Tables of Interatomic Distances and Configuration in Molecules and Ions*. Suppl. 1956–59. London: The Chemical Society.

*Acta Cryst.* (1978). **B34**, 562–567

## The Crystal Structure of 3-(*p*-Chlorophenyl)-4-oxo-3a,5,6,6a-tetraphenyl-3a,4-dihydrocyclopenta[2,3-*d*]isoxazoline

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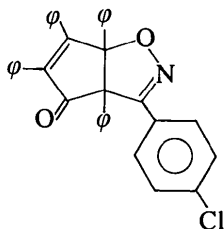
The crystal structure of 3-(*p*-chlorophenyl)-4-oxo-3a,5,6,6a-tetraphenyl-3a,4-dihydrocyclopenta[2,3-*d*]isoxazoline,  $C_{36}H_{24}NO_2Cl$ , has been determined from three-dimensional intensities, measured with an automated Philips PW 1100 single-crystal diffractometer (1603 independent non-zero reflexions). The structure was solved by direct phase determination with *MULTAN*. The cell constants, obtained by least-squares calculations from direct  $\theta$ -value measurements on the diffractometer, are:  $a = 14.373$  (4),  $b = 21.084$  (7),  $c = 9.261$  (3) Å,  $\beta = 99.27$  (3)°,  $Z = 4$ ; the space group is  $P2_1/n$ . 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  $R = 0.060$ . Correction for anomalous scattering of the Cl atom was applied. A molecule of the compound has a cyclopentenone–isoxazoline structure. To the two central five-membered isoxazoline and cyclopentenone rings, at an angle of 107°, are linked five benzene rings with different orientations. A characteristic feature is the coplanar system formed by the isoxazoline ring and the *p*-chloro-substituted benzene ring.

### Introduction

Reactions yielding isoxazoline derivatives are of considerable theoretical interest as regards regioselectivity owing to the influence of the frontier molecular orbitals

of the reacting systems. To this end a series of new compounds were prepared at the Laboratory of Organic Chemistry, Aristotle University of Thessaloniki (Alexandrou & Argyropoulos, 1977). Since chemical and spectroscopic data were in most cases

inconclusive as regards the structure and conformation of the molecules, a systematic X-ray analysis was considered necessary. The structure determination of 3-(*p*-chlorophenyl)-4-oxo-3a,5,6,6a-tetraphenyl-3a,4-dihydrocyclopenta[2,3-*d*]isoxazoline (CPCI in the following)



is the first of a series.

### Experimental

Pure, colourless CPCI crystals were kindly provided by Professor N. E. Alexandrou and Mr N. Argyropoulos. The crystals are monoclinic plates. A transparent single crystal, with dimensions 0.20 × 0.25 × 0.35 mm, was selected and centred on our computer-controlled Philips PW 1100 four-circle single-crystal diffractometer. With Mo  $K\alpha$  radiation ( $\lambda = 0.71069 \text{ \AA}$ ) the  $\theta$  angles of 200 strong reflexions with large  $\theta$  values were directly measured on the diffractometer and subsequently processed with the least-squares program *PARAM* [part of the XRAY system of crystallographic programs (Stewart, Kruger, Ammon, Dickinson & Hall, 1976)]. The final values of the cell parameters are included in Table 1. 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  $\omega$  scan mode, using graphite-monochromatized Mo  $K\alpha$  radiation. The intensities of 3149 independent reflexions up to  $2\theta = 45^\circ$  (maximum  $h, k, l = 12, 18, \pm 8$  respectively) were examined and measured. Of these, 1603 with intensities greater than  $2\sigma$  were considered as observed and included in all subsequent computations.

Integrated intensities were converted to  $|F_o|$  values in the usual way, using the special measurement treatment program *DATRED* (Main, 1970). Since  $\mu$  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.

### Determination of the structure and refinement

The structure was essentially solved by direct phase determination with the *MULTAN* system. The phases of 280 strong reflexions were determined and on the

Table 1. *Crystal data for CPCI*

Standard errors, given in parentheses, refer to last digit.	
$C_{36}H_{24}NO_2Cl$	FW 538.05
Monoclinic	$Z = 4$
Space group $P2_1/n$	$F(000) = 1120$
$a = 14.373 (4) \text{ \AA}$	$\rho_{\text{calc}} = 1.290 \text{ g cm}^{-3}$
$b = 21.084 (7)$	$\rho_{\text{meas}} = 1.303$
$c = 9.261 (3)$	m.p. 213–214 °C
$\beta = 99.27 (3)^\circ$	$\mu = 1.78 \text{ cm}^{-1}$
$V = 2769.91 \text{ \AA}^3$	$\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$

Table 2. *Atomic coordinates and equivalent isotropic temperature factors for the non-hydrogen atoms in CPCI, with standard deviations in parentheses*

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å <sup>2</sup> )
C(1)	0.6893 (2)	0.4377 (1)	0.9638 (3)	4.94
O(1)	0.1738 (4)	0.3699 (3)	0.1739 (6)	2.99
O(2)	0.4077 (4)	0.4672 (3)	0.5135 (7)	3.50
N(1)	0.2508 (5)	0.3821 (4)	0.0986 (8)	3.01
C(1)	0.3262 (6)	0.3945 (4)	0.1913 (9)	2.48
C(2)	0.3122 (6)	0.3908 (4)	0.3521 (9)	2.36
C(3)	0.3355 (5)	0.4548 (4)	0.4313 (8)	2.50
C(4)	0.2510 (5)	0.4967 (4)	0.3946 (8)	2.27
C(5)	0.1760 (5)	0.4620 (4)	0.3350 (8)	2.07
C(6)	0.2014 (5)	0.3904 (4)	0.3255 (8)	2.34
C(7)	0.4158 (5)	0.4067 (4)	0.1361 (8)	2.79
C(8)	0.4170 (6)	0.4086 (4)	−0.0150 (9)	3.19
C(9)	0.5007 (6)	0.4193 (4)	−0.0689 (9)	3.28
C(10)	0.5829 (6)	0.4261 (4)	0.0301 (10)	3.27
C(11)	0.5839 (6)	0.4228 (4)	0.1788 (9)	3.62
C(12)	0.4987 (6)	0.4145 (4)	0.2317 (8)	2.74
C(13)	0.3583 (5)	0.3323 (4)	0.4286 (8)	2.35
C(14)	0.3733 (6)	0.2729 (4)	0.3463 (10)	4.08
C(15)	0.4087 (7)	0.2227 (5)	0.4141 (12)	5.31
C(16)	0.4268 (7)	0.2195 (5)	0.5673 (13)	5.42
C(17)	0.4108 (7)	0.2717 (5)	0.6503 (10)	5.12
C(18)	0.3767 (6)	0.3278 (4)	0.5810 (9)	3.69
C(19)	0.2562 (5)	0.5658 (4)	0.4335 (7)	2.53
C(20)	0.3389 (6)	0.6009 (4)	0.4253 (9)	3.18
C(21)	0.3462 (9)	0.6658 (5)	0.4590 (11)	4.40
C(22)	0.2704 (9)	0.6952 (6)	0.5043 (11)	5.27
C(23)	0.1879 (7)	0.6624 (5)	0.5142 (10)	5.50
C(24)	0.1809 (6)	0.5985 (4)	0.4808 (9)	3.35
C(25)	0.8005 (5)	0.4839 (3)	0.2691 (8)	2.06
C(26)	−0.0004 (6)	0.4504 (4)	0.2848 (9)	3.03
C(27)	−0.0896 (6)	0.4707 (4)	0.2197 (10)	4.15
C(28)	−0.0997 (6)	0.5250 (4)	0.1372 (9)	3.48
C(29)	−0.0200 (6)	0.5599 (4)	0.1172 (8)	3.23
C(30)	0.0687 (6)	0.5388 (4)	0.1843 (8)	3.10
C(31)	0.1572 (5)	0.3472 (4)	0.4275 (9)	2.64
C(32)	0.1458 (6)	0.3666 (4)	0.5676 (10)	4.39
C(33)	0.1160 (8)	0.3236 (5)	0.6653 (11)	6.31
C(34)	0.0946 (8)	0.2634 (5)	0.6201 (14)	6.19
C(35)	0.1047 (8)	0.2432 (5)	0.4829 (14)	6.00
C(36)	0.1365 (6)	0.2858 (4)	0.3854 (10)	4.17

resulting *E* map it was possible to locate all the non-hydrogen atoms of the asymmetric unit. A structure factor calculation at this stage, with an overall  $B = 2.95 \text{ \AA}^2$ , gave  $R = 0.214$ .

Table 3. Atomic coordinates and isotropic temperature factors for the hydrogen atoms in CPCI

	x	y	z	B (Å <sup>2</sup> )
H(C8)	0.359 (5)	0.405 (4)	-0.082 (8)	5.8 (2.0)
H(C9)	0.497 (6)	0.427 (4)	-0.185 (9)	5.9 (2.3)
H(C11)	0.644 (4)	0.432 (3)	0.251 (7)	3.6 (1.6)
H(C12)	0.499 (4)	0.414 (3)	0.341 (7)	3.2 (1.6)
H(C14)	0.362 (6)	0.281 (4)	0.235 (9)	7.1 (2.4)
H(C15)	0.414 (6)	0.182 (4)	0.347 (8)	6.2 (2.2)
H(C16)	0.447 (7)	0.180 (5)	0.611 (11)	8.7 (3.0)
H(C17)	0.423 (5)	0.272 (3)	0.765 (8)	5.5 (1.9)
H(C18)	0.365 (5)	0.364 (3)	0.643 (7)	3.2 (2.2)
H(C20)	0.393 (5)	0.575 (3)	0.393 (7)	2.3 (1.7)
H(C21)	0.412 (7)	0.686 (5)	0.444 (10)	7.5 (2.8)
H(C22)	0.277 (6)	0.739 (4)	0.525 (9)	2.6 (2.3)
H(C23)	0.131 (6)	0.687 (4)	0.535 (10)	6.5 (2.6)
H(C24)	0.122 (4)	0.572 (3)	0.486 (7)	2.2 (1.6)
H(C26)	0.005 (4)	0.407 (2)	0.344 (6)	1.4 (1.2)
H(C27)	-0.147 (5)	0.443 (3)	0.241 (7)	3.5 (1.7)
H(C28)	-0.162 (5)	0.542 (3)	0.090 (7)	4.2 (1.9)
H(C29)	-0.033 (5)	0.600 (3)	0.047 (8)	3.9 (1.8)
H(C30)	0.126 (4)	0.562 (3)	0.164 (6)	1.0 (1.2)
H(C32)	0.168 (6)	0.409 (4)	0.605 (9)	6.4 (2.4)
H(C33)	0.108 (6)	0.338 (4)	0.764 (10)	7.9 (2.6)
H(C34)	0.071 (6)	0.233 (4)	0.688 (9)	5.7 (2.3)
H(C35)	0.089 (7)	0.199 (4)	0.450 (10)	7.4 (2.7)
H(C36)	0.143 (5)	0.272 (4)	0.282 (9)	5.2 (2.2)

Table 4. Interatomic distances (Å) in CPCI, with their standard deviations in parentheses

O(1)-N(1)	1.423 (10)	C(13)-C(14)	1.397 (11)
N(1)-C(1)	1.295 (11)	C(14)-C(15)	1.397 (14)
C(1)-C(2)	1.537 (12)	C(15)-C(16)	1.382 (15)
C(1)-C(7)	1.484 (12)	C(16)-C(17)	1.402 (16)
C(2)-C(6)	1.573 (11)	C(17)-C(18)	1.398 (13)
C(2)-C(13)	1.521 (11)	C(18)-C(13)	1.396 (12)
C(6)-C(31)	1.522 (11)		
C(6)-O(1)	1.462 (9)	C(19)-C(20)	1.412 (12)
		C(20)-C(21)	1.405 (14)
C(2)-C(3)	1.546 (11)	C(21)-C(22)	1.375 (17)
C(3)-O(2)	1.213 (9)	C(22)-C(23)	1.388 (16)
C(3)-C(4)	1.496 (10)	C(23)-C(24)	1.382 (13)
C(4)-C(5)	1.346 (10)	C(24)-C(19)	1.411 (12)
C(4)-C(19)	1.498 (11)		
C(5)-C(6)	1.558 (10)	C(25)-C(26)	1.393 (10)
C(5)-C(25)	1.483 (9)	C(26)-C(27)	1.398 (11)
		C(27)-C(28)	1.399 (12)
C(7)-C(8)	1.403 (11)	C(28)-C(29)	1.370 (12)
C(8)-C(9)	1.394 (12)	C(29)-C(30)	1.394 (11)
C(9)-C(10)	1.381 (11)	C(30)-C(25)	1.389 (11)
C(10)-Cl(1)	1.755 (9)		
C(10)-C(11)	1.377 (12)	C(31)-C(32)	1.395 (13)
C(11)-C(12)	1.402 (12)	C(32)-C(33)	1.397 (15)
C(12)-C(7)	1.374 (10)	C(33)-C(34)	1.357 (16)
		C(34)-C(35)	1.369 (18)
		C(35)-C(36)	1.402 (15)
		C(36)-C(31)	1.371 (12)

Refinement of the structure was carried out by full-matrix least-squares calculations. The atomic scattering factors for Cl, O, N and C were from Cromer & Waber (1965), and for H from Stewart, Davidson & Simpson

Table 5. Bond angles (°) in CPCI, with their standard deviations in parentheses

C(6)-O(1)-N(1)	107.8 (0.8)	C(2)-C(13)-C(14)	119.6 (0.7)
O(1)-N(1)-C(1)	110.7 (0.8)	C(2)-C(13)-C(18)	121.6 (0.7)
N(1)-C(1)-C(2)	114.0 (0.8)	C(14)-C(13)-C(18)	118.5 (0.7)
N(1)-C(1)-C(7)	119.1 (0.8)	C(13)-C(14)-C(15)	121.0 (0.8)
C(2)-C(1)-C(7)	126.8 (0.7)	C(14)-C(15)-C(16)	119.3 (0.9)
C(1)-C(2)-C(6)	97.8 (0.6)	C(15)-C(16)-C(17)	120.3 (0.9)
C(1)-C(2)-C(13)	112.2 (0.7)	C(16)-C(17)-C(18)	119.7 (0.9)
C(1)-C(2)-C(3)	111.4 (0.7)	C(17)-C(18)-C(13)	121.0 (0.8)
C(13)-C(2)-C(6)	115.0 (0.7)		
C(2)-C(6)-C(31)	114.8 (0.6)	C(4)-C(19)-C(20)	120.4 (0.7)
C(2)-C(6)-O(1)	105.2 (0.6)	C(20)-C(19)-C(24)	117.0 (0.7)
C(5)-C(6)-O(1)	108.1 (0.5)	C(19)-C(20)-C(21)	122.2 (0.9)
C(31)-C(6)-O(1)	110.1 (0.6)	C(20)-C(21)-C(22)	118.1 (1.0)
		C(21)-C(22)-C(23)	121.5 (1.0)
C(3)-C(2)-C(6)	102.4 (0.6)	C(22)-C(23)-C(24)	120.2 (1.0)
C(3)-C(2)-C(13)	116.3 (0.6)	C(23)-C(24)-C(19)	120.9 (0.8)
C(2)-C(3)-C(4)	107.6 (0.6)	C(24)-C(19)-C(4)	122.6 (0.7)
C(2)-C(3)-O(2)	125.7 (0.7)		
O(2)-C(3)-C(4)	126.6 (0.7)	C(5)-C(25)-C(26)	122.2 (0.6)
C(3)-C(4)-C(5)	109.8 (0.6)	C(5)-C(25)-C(30)	120.4 (0.6)
C(3)-C(4)-C(19)	120.8 (0.6)	C(25)-C(26)-C(27)	121.6 (0.7)
C(19)-C(4)-C(5)	129.3 (0.7)	C(26)-C(27)-C(28)	118.8 (0.7)
C(4)-C(5)-C(6)	111.8 (0.6)	C(27)-C(28)-C(29)	119.9 (0.7)
C(4)-C(5)-C(25)	128.8 (0.7)	C(28)-C(29)-C(30)	120.4 (0.8)
C(25)-C(5)-C(6)	119.2 (0.6)	C(29)-C(30)-C(25)	122.3 (0.7)
C(5)-C(6)-C(2)	103.1 (0.6)	C(30)-C(25)-C(26)	117.0 (0.6)
C(5)-C(6)-C(31)	114.8 (0.6)		
		C(6)-C(31)-C(36)	118.8 (0.7)
C(1)-C(7)-C(8)	119.9 (0.6)	C(6)-C(31)-C(32)	121.6 (0.7)
C(1)-C(7)-C(12)	120.6 (0.7)	C(31)-C(32)-C(33)	120.4 (0.8)
C(8)-C(7)-C(12)	119.4 (0.7)	C(32)-C(33)-C(34)	119.1 (1.0)
C(7)-C(8)-C(9)	120.7 (0.7)	C(33)-C(34)-C(35)	121.7 (1.1)
C(8)-C(9)-C(10)	118.3 (0.7)	C(34)-C(35)-C(36)	119.5 (0.9)
C(9)-C(10)-Cl	118.8 (0.7)	C(35)-C(36)-C(31)	120.1 (0.9)
Cl-C(10)-C(11)	119.2 (0.6)	C(36)-C(31)-C(32)	119.2 (0.7)
C(9)-C(10)-C(11)	121.9 (0.8)		
C(10)-C(11)-C(12)	119.2 (0.7)		
C(11)-C(12)-C(7)	120.3 (0.7)		

(1965). Anomalous-dispersion corrections for Cl were taken from *International Tables for X-ray Crystallography* (1968). A single scale factor was used for the whole set of reflexion data.

With the same overall temperature factor and unit weights, refinement reduced *R* to 0.136 in two cycles. Two further cycles with isotropic temperature factors lowered *R* to 0.113. The four subsequent cycles with anisotropic temperature coefficients gave *R* = 0.095. The H atoms were then located with the help of the XRAY system, and included in four 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 0.065. At this stage a systematic weight analysis was carried out, which led to the following weighting scheme:  $w = (|F_o|/18)^2$  for  $|F_o| < 18$ ,  $w = 1$  for  $18 < |F_o| < 133$  and  $w = (133/|F_o|)^2$  for  $|F_o| > 133$ . Convergence was reached at *R* = 0.060 (*R<sub>w</sub>* = 0.057) in two further cycles. The average shift/error ratio of the last cycle was less than 0.3.



and  $O(2)-C(20) = 3.054(11) \text{ \AA}$ . It occupies the apex of a flat trigonal pyramid, whose base, an almost equilateral triangle, is formed by these C atoms. The three benzene rings  $B_1$ ,  $B_2$  and  $B_3$  lie almost symmetrically with respect to the line  $C(3)-O(2)$ .

The geometrical features of the benzene rings are in good agreement with the usually accepted values. Mean bond lengths of the rings  $B_1$ ,  $B_2$ ,  $B_3$ ,  $B_4$  and  $B_5$  are 1.388 (11), 1.387 (13), 1.396 (14), 1.391 (11) and 1.382 (15)  $\text{\AA}$  respectively.

The  $Cl-C_{ar}$  bond length, 1.755 (9)  $\text{\AA}$ , and the corresponding endocyclic angle,  $\alpha = 121.9(0.8)^\circ$ , compare well with the mean values of 1.7404 (11)  $\text{\AA}$  and  $121.37(9)^\circ$  deduced by Domenicano, Vaciano & Coulson (1975) for a number of *p*-substituted chlorobenzenes. The  $Cl-C_{ar}$  bond is bent by an angle of  $10.6^\circ$  out of the plane of the benzene ring  $B_1$ , the distance of the Cl atom from the plane being 0.322  $\text{\AA}$ . This rather large bending may be attributed to the

requirement that intramolecular non-bonded interactions should be minimized.

The general features of the five-membered isoxazoline and cyclopentenone rings are normal. The  $C(3)=O$  bond distance of the cyclopentenone ring is 1.213 (9)  $\text{\AA}$ . Owing to the conjugation  $O=C(3)-C(4)=C(5)$ , the double-bond distance  $C(3)=O$ , 1.213  $\text{\AA}$ , appears shortened and close to the usually accepted mean value of 1.207 (6)  $\text{\AA}$  (*International Tables*, 1968) in conjugated systems. The short  $C(3)-C(4)$  bond length, 1.496 (10)  $\text{\AA}$ , of the conjugated system is quite normal.

The intramolecular contact distances  $O(2)-H(C12) = 2.49(7) \text{ \AA}$ ,  $O(2)-H(C18) = 2.61(7) \text{ \AA}$  and  $O(2)-H(C20) = 2.53(7) \text{ \AA}$  almost coincide with the sum of the van der Waals radii of the corresponding atoms. The distances  $Cl-H(C9) = 2.89(8) \text{ \AA}$  and  $Cl-H(C11) = 2.84(7) \text{ \AA}$  are slightly shorter than the calculated distances. All other contact distances are within the expected values.

Fig. 2 is a clinographic projection of the structure showing the molecular packing of CPCl in the unit cell. The various intermolecular distances are normal. Some interesting intermolecular distances are shown in Table 7. They compare well with the sum of the van der Waals radii of the corresponding atoms.

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Table 7. Intermolecular distances ( $\text{\AA}$ )

$Cl \cdots H(C27)^I$	3.20 (6)
$Cl \cdots H(C28)^I$	3.16 (7)
$Cl \cdots H(C30)^{III}$	3.08 (6)
$O(2) \cdots H(C12)^{II}$	3.05 (6)
$O(2) \cdots H(C20)^{II}$	3.00 (8)

Symmetry code

(i)	$1 + x,$	$y,$	$1 + z$
(ii)	$x,$	$y,$	$1 + z$
(iii)	$1 - x,$	$1 - y,$	$1 - z$

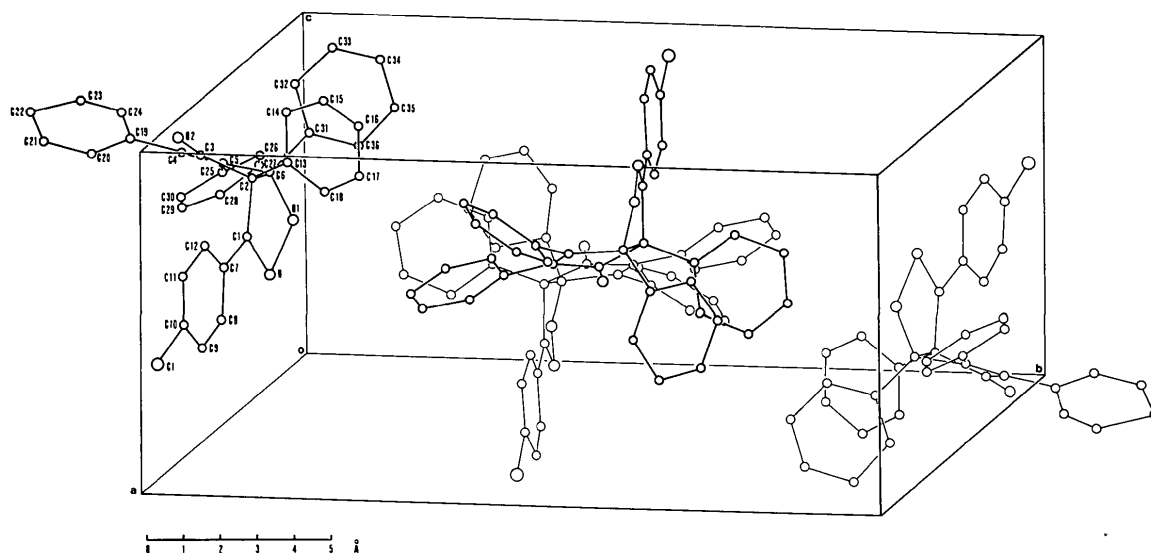


Fig. 2. Clinographic projection of the unit cell, showing the molecular packing.

## References

- ALEXANDROU, N. E. & ARGYROPOULOS, N. (1977). Sixth International Congress of Heterocyclic Chemistry, Teheran.
- CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.
- DOMENICANO, A., VACIAGO, A. & COULSON, C. A. (1975). *Acta Cryst.* **B31**, 1630–1641.
- International Tables for X-ray Crystallography* (1968). Vol. III, 2nd edition, pp. 215–216. Birmingham: Kynoch Press.
- MAIN, P. (1970). Private communication.
- MAIN, P., WOOLFSON, 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.
- STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. & HALL, S. R. (1976). The XRAY 76 system. Tech. Rep. 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.

*Acta Cryst.* (1978). **B34**, 567–578

## An 11 Å-Resolution Electron Density Map of Southern Bean Mosaic Virus

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Screened precession photography was used to collect 11 Å-resolution data for native and K<sub>2</sub>HgI<sub>4</sub> derivative crystals of southern bean mosaic virus. Rotation functions showed that not only the virus but also the sites of HgI<sub>4</sub><sup>2-</sup> attachment had icosahedral symmetry. A difference Patterson synthesis was systematically searched for icosahedral distributions of vectors between heavy atoms. The resultant search function was examined for sets of solutions consistent with  $T=3$  symmetry. The major set of consistent peaks was then refined by a least-squares procedure which assumed the non-crystallographic symmetry. The resultant single isomorphous replacement phases were used to compute an electron density distribution which was icosahedrally averaged. The averaged map was the basis for an improved set of phases. Two further cycles of molecular replacement produced an electron density map with features similar to an earlier 22.5 Å-resolution structure which had been determined without the benefit of any heavy-atom derivatives. The new 11 Å-resolution map showed additional details consistent with the  $T=3$  symmetry.

### Introduction

Particles of southern bean mosaic virus (SBMV) are roughly spherical with a molecular weight of  $6.6 \times 10^6$  (Miller & Price, 1946; Yphantis, 1964) (Appendix I).

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Their mean diameter is 284 Å, although the distance between opposing fivefold vertices is 318 Å (Johnson, Akimoto, Suck, Rayment & Rossmann, 1976). There are 180 identical protein subunits, each of molecular weight 28 250 (Tremaine, 1966; Hill & Shepherd, 1971) (Appendix I) consistent with  $T=3$  quasi-symmetry (Caspar & Klug, 1962). Four different crystal forms have been reported (Akimoto, Wagner, Johnson & Rossmann, 1975), of which the type II crystals are the most suitable for detailed structural investigations.