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The Crystal Structure of 3a,5-Bis(methoxycarbonyl)-3-(*p*-nitrophenyl)-4-oxo-6,6a-diphenyl-3a,4-dihydrocyclopenta[2,3-*d*]isoxazoline

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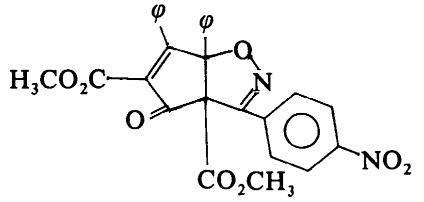
The crystal structure of the title compound, $C_{28}H_{20}N_2O_8$, has been determined from three-dimensional intensities, measured with an automated Philips PW 1100 single-crystal diffractometer (1274 independent observed reflexions). The structure was solved by direct phase determination with *MULTAN*. The cell constants of the reduced cell, obtained by least-squares calculations from direct θ-value measurements on the diffractometer, are: $a = 12{\cdot}576\ (4)$, $b = 10{\cdot}014\ (3)$, $c = 10{\cdot}492\ (4)$ Å, $\alpha = 91{\cdot}66\ (3)$, $\beta = 95{\cdot}52\ (3)$, $\gamma = 88{\cdot}46\ (3)°$, $Z = 2$; the space group is $P\bar{1}$. 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 $R = 0{\cdot}082$. A molecule of the compound has a cyclopentenone–isoxazoline structure. To the two central five-membered isoxazoline and cyclopentenone rings, at an angle of 110·4°, are linked three benzene rings and two methoxycarbonyl groups with different orientations. A characteristic feature is the roughly coplanar system formed by the isoxazoline ring and the *p*-NO_2-substituted benzene ring.

Introduction

A number of new isoxazoline derivatives with theoretical interest have been prepared at the Laboratory of Organic Chemistry of the Aristotle University of Thessaloniki (Alexandrou & Argyropoulos, 1977). The need for dependable structural and conformational information necessitated a systematic X-ray analysis of these compounds. The structure determination of 3a,5-bis(methoxycarbonyl)-3-(*p*-nitrophenyl)-4-oxo-6,6a-diphenyl-3a,4-dihydrocyclopenta[2,3-*d*]isoxazoline (NPCCI) is the second of a series under investigation (*cf.* Rodiou, Kokkou & Rentzeperis, 1978).



Experimental

Pure, colourless NPCCI crystals were kindly provided by Professor N. E. Alexandrou and Mr N. Argyropoulos. The crystals are triclinic prisms. A transparent single crystal, with dimensions 0·22 × 0·26 × 0·30 mm, was selected and centred on our computer-controlled Philips PW 1100 four-circle single-crystal diffractometer. With Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ 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)]. The final values for the reduced cell are given in Table 1. Intensity statistics indicated the presence of a symmetry centre and so the space group $P\bar{1}$ was assumed; this was confirmed by the determined structure. 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 monochromatized with a graphite monochromator. The intensities of 3380 independent reflexions up to $2\theta = 45°$ (max $hkl = 13, \pm 10, \pm 10$ respectively) were examined and measured. Of these, 1274 with intensities greater than 2σ were considered as observed and included in all subsequent computations.

Integrated intensities were converted to F_o values in

Table 1. *Crystal data for NPCCI*

Standard errors, given in parentheses, refer to the last digit.

$C_{28}H_{20}N_2O_8$
Triclinic
$a = 12{\cdot}576\ (4)$ Å
$b = 10{\cdot}014\ (3)$
$c = 10{\cdot}492\ (4)$
$\alpha = 91{\cdot}66\ (3)°$
$\beta = 95{\cdot}52\ (3)$
$\gamma = 88{\cdot}46\ (3)$
$V = 1314{\cdot}31$ Å^3

FW 512·456
Space group $P\bar{1}$
$Z = 2$
$F(000) = 532$
$\rho_{\rm calc} = 1{\cdot}266$ g cm^{-3}
$\rho_{\rm meas} = 1{\cdot}300$
m.p. 158–159 °C
$\mu = 1{\cdot}0493$ cm^{-1}
λ(Mo $K\alpha$) = 0·71069 Å

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.

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 resulting E map it was possible to locate 37 of the 38 non-hydrogen atoms of the asymmetric unit. A structure factor calculation at this stage, with the 280 reflexions and an overall $B = 2\cdot43$ Å^2, gave $R = 0\cdot224$. The missing C atom was easily located on Fourier and difference Fourier maps.

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

	x	*y*	*z*	*B* (Å^2)
O(1)	0·7826 (6)	0·2433 (7)	0·8971 (7)	3·0
O(2)	0·7488 (7)	0·9095 (9)	0·5826 (9)	4·4
O(3)	0·3650 (10)	0·5815 (13)	0·8225 (13)	9·0
O(4)	0·4150 (8)	0·5889 (9)	0·0257 (11)	6·0
O(5)	0·6288 (7)	0·1618 (9)	0·5048 (8)	5·2
O(6)	0·5552 (6)	0·2314 (8)	0·6834 (7)	4·0
O(7)	0·9876 (7)	0·8627 (9)	0·5694 (8)	5·1
O(8)	0·0569 (8)	0·9073 (12)	0·7685 (9)	8·5
N(1)	0·7219 (8)	0·1419 (10)	0·9370 (9)	2·9
N(2)	0·4148 (11)	0·6296 (13)	0·9135 (14)	4·9
C(1)	0·6844 (9)	0·0670 (13)	0·8412 (12)	2·7
C(2)	0·7200 (8)	0·1105 (10)	0·7140 (11)	1·9
C(3)	0·7891 (11)	0·9997 (13)	0·6500 (12)	3·3
C(4)	0·9023 (8)	0·0235 (11)	0·6977 (10)	2·5
C(5)	0·9121 (8)	0·1456 (11)	0·7631 (10)	1·9
C(6)	0·8036 (9)	0·2176 (10)	0·7641 (10)	2·0
C(7)	0·6148 (10)	0·9522 (12)	0·8568 (11)	3·3
C(8)	0·5482 (10)	0·8986 (13)	0·7555 (11)	3·8
C(9)	0·4791 (10)	0·7960 (14)	0·7737 (12)	4·0
C(10)	0·4855 (9)	0·7451 (13)	0·8932 (13)	3·7
C(11)	0·5484 (10)	0·7920 (12)	0·9979 (12)	4·3
C(12)	0·6150 (12)	0·8999 (13)	0·9780 (13)	4·3
C(13)	0·6309 (9)	0·1664 (11)	0·6207 (11)	3·2
C(14)	0·4693 (11)	0·2989 (16)	0·6052 (14)	6·6
C(15)	0·9866 (9)	0·9199 (12)	0·6711 (11)	3·3
C(16)	0·1476 (15)	0·8154 (23)	0·7484 (16)	11·5
C(17)	0·0108 (9)	0·2108 (13)	0·8159 (13)	3·9
C(18)	0·0192 (14)	0·2652 (22)	0·9391 (19)	10·3
C(19)	0·1107 (14)	0·3364 (23)	0·9849 (23)	11·9
C(20)	0·1872 (15)	0·3548 (20)	0·9089 (27)	9·1
C(21)	0·1841 (18)	0·2968 (30)	0·7914 (21)	10·9
C(22)	0·0893 (13)	0·2269 (22)	0·7449 (16)	9·4
C(23)	0·8002 (9)	0·3482 (12)	0·6957 (12)	2·8
C(24)	0·7488 (11)	0·4608 (14)	0·7404 (14)	4·2
C(25)	0·7454 (13)	0·5811 (15)	0·6780 (17)	5·7
C(26)	0·7925 (13)	0·5890 (15)	0·5653 (14)	5·0
C(27)	0·8470 (11)	0·4821 (15)	0·5145 (13)	4·9
C(28)	0·8498 (10)	0·3617 (14)	0·5844 (13)	4·1

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, refinement reduced R to 0·183 in three cvcles.

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

	x	*y*	*z*	*B* (Å^2)
H(C8)	0·551 (8)	0·946 (9)	0·675 (9)	3·8 (3·5)
H(C9)	0·435 (8)	0·755 (9)	0·707 (9)	3·6 (3·4)
H(C11)	0·566 (7)	0·759 (9)	0·064 (9)	3·0 (2·9)
H(C12)	0·651 (7)	0·933 (9)	0·039 (9)	2·9 (2·5)
H1(C14)	0·511 (10)	0·364 (12)	0·563 (12)	9·2 (3·7)
H2(C14)	0·416 (8)	0·352 (9)	0·667 (9)	5·6 (2·6)
H3(C14)	0·436 (8)	0·247 (10)	0·514 (10)	5·4 (2·9)
H1(C16)	0·121 (15)	0·716 (16)	0·719 (18)	16·0 (5·0)
H2(C16)	0·185 (14)	0·841 (17)	0·690 (20)	14·9 (4·9)
H3(C16)	0·188 (11)	0·833 (14)	0·816 (13)	11·1 (4·6)
H(C18)	0·975 (10)	0·238 (13)	0·986 (13)	2·7 (2·5)
H(C19)	0·114 (14)	0·351 (16)	0·083 (16)	11·7 (5·6)
H(C20)	0·252 (9)	0·404 (11)	0·927 (10)	6·6 (3·2)
H(C21)	0·227 (9)	0·352 (12)	0·725 (11)	8·5 (3·3)
H(C22)	0·115 (10)	0·184 (13)	0·672 (12)	9·4 (3·9)
H(C24)	0·706 (6)	0·460 (8)	0·810 (8)	1·8 (1·5)
H(C25)	0·709 (7)	0·648 (9)	0·685 (9)	3·1 (2·5)
H(C26)	0·781 (9)	0·670 (11)	0·513 (10)	7·4 (3·2)
H(C27)	0·903 (9)	0·496 (11)	0·443 (10)	7·3 (3·2)
H(C28)	0·902 (7)	0·302 (8)	0·552 (8)	2·8 (2·2)

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

O(1)–N(1)	1·38 (1)	C(13)–O(5)	1·21 (1)
N(1)–C(1)	1·30 (1)	C(13)–O(6)	1·35 (1)
C(1)–C(2)	1·53 (2)	O(6)–C(14)	1·46 (2)
C(2)–C(6)	1·57 (1)		
C(6)–O(1)	1·46 (1)	C(15)–O(7)	1·20 (1)
C(1)–C(7)	1·49 (1)	C(15)–O(8)	1·29 (1)
C(2)–C(13)	1·52 (1)	O(8)–C(16)	1·47 (2)
C(6)–C(23)	1·51 (2)		
		C(17)–C(18)	1·38 (2)
C(2)–C(3)	1·57 (2)	C(18)–C(19)	1·41 (3)
C(3)–C(4)	1·49 (2)	C(19)–C(20)	1·33 (3)
C(4)–C(5)	1·39 (2)	C(20)–C(21)	1·34 (4)
C(5)–C(6)	1·53 (1)	C(21)–C(22)	1·44 (3)
C(3)–O(2)	1·22 (2)	C(22)–C(17)	1·31 (2)
C(4)–C(15)	1·50 (2)		
C(5)–C(17)	1·47 (2)	C(23)–C(24)	1·38 (2)
		C(24)–C(25)	1·39 (2)
C(7)–C(8)	1·39 (2)	C(25)–C(26)	1·38 (2)
C(8)–C(9)	1·39 (2)	C(26)–C(27)	1·38 (2)
C(9)–C(10)	1·36 (2)	C(27)–C(28)	1·43 (2)
C(10)–C(11)	1·37 (2)	C(28)–C(23)	1·39 (2)
C(11)–C(12)	1·42 (2)		
C(12)–C(7)	1·39 (2)		
C(10)–N(2)	1·51 (2)		
N(2)–O(3)	1·19 (2)		
N(2)–O(4)	1·25 (2)		

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

C(6)–O(1)–N(1)	109·6 (0·8)	C(3)–C(2)–C(6)	103·5 (0·8)	C(1)–C(7)–C(8)	122·3 (1·1)
O(1)–N(1)–C(1)	111·4 (0·9)	C(3)–C(2)–C(13)	112·3 (0·9)	C(1)–C(7)–C(12)	118·1 (1·0)
N(1)–C(1)–C(2)	112·4 (0·9)	C(2)–C(3)–C(4)	106·7 (0·9)	C(8)–C(7)–C(12)	119·6 (1·2)
N(1)–C(1)–C(7)	122·6 (1·1)	C(2)–C(3)–O(2)	122·1 (1·1)	C(7)–C(8)–C(9)	121·4 (1·2)
C(2)–C(1)–C(7)	125·0 (1·0)	O(2)–C(3)–C(4)	130·9 (1·2)	C(8)–C(9)–C(10)	116·6 (1·1)
C(1)–C(2)–C(6)	99·8 (0·8)	C(3)–C(4)–C(5)	110·9 (1·0)	C(9)–C(10)–C(11)	125·6 (1·2)
C(1)–C(2)–C(13)	114·8 (0·9)	C(3)–C(4)–C(15)	119·3 (1·0)	C(9)–C(10)–N(2)	117·4 (1·2)
C(1)–C(2)–C(3)	112·2 (0·9)	C(15)–C(4)–C(5)	129·7 (1·0)	N(2)–C(10)–C(11)	117·0 (1·2)
C(13)–C(2)–C(6)	113·0 (0·9)	C(4)–C(5)–C(17)	127·9 (1·0)	O(3)–N(2)–O(4)	124·6 (1·4)
C(2)–C(6)–C(23)	117·6 (0·9)	C(4)–C(5)–C(6)	110·8 (0·9)	O(3)–N(2)–C(10)	118·1 (1·4)
C(2)–C(6)–O(1)	104·1 (0·8)	C(17)–C(5)–C(6)	121·0 (0·9)	O(4)–N(2)–C(10)	117·3 (1·2)
C(5)–C(6)–O(1)	108·3 (0·8)	C(5)–C(6)–C(2)	105·0 (0·8)	C(10)–C(11)–C(12)	116·6 (1·2)
C(23)–C(6)–O(1)	109·2 (0·8)	C(5)–C(6)–C(23)	112·1 (0·9)	C(11)–C(12)–C(7)	120·1 (1·2)
C(2)–C(13)–O(5)	125·8 (1·1)	C(5)–C(17)–C(18)	120·4 (1·3)	C(6)–C(23)–C(24)	122·2 (1·1)
C(2)–C(13)–O(6)	111·2 (0·9)	C(5)–C(17)–C(22)	120·9 (1·3)	C(6)–C(23)–C(28)	121·6 (1·1)
O(5)–C(13)–O(6)	122·9 (1·0)	C(18)–C(17)–C(22)	118·5 (1·4)	C(24)–C(23)–C(28)	116·3 (1·2)
C(13)–O(6)–C(14)	116·8 (0·9)	C(17)–C(18)–C(19)	119·8 (1·8)	C(23)–C(24)–C(25)	122·4 (1·4)
		C(18)–C(19)–C(20)	120·1 (2·2)	C(24)–C(25)–C(26)	119·5 (1·4)
C(4)–C(15)–O(7)	122·3 (1·0)	C(19)–C(20)–C(21)	121·3 (2·1)	C(25)–C(26)–C(27)	122·1 (1·4)
C(4)–C(15)–O(8)	111·3 (1·0)	C(20)–C(21)–C(22)	117·7 (2·0)	C(26)–C(27)–C(28)	115·9 (1·3)
O(7)–C(15)–O(8)	126·4 (1·1)	C(21)–C(22)–C(17)	122·3 (1·7)	C(27)–C(28)–C(23)	123·8 (1·2)
C(15)–O(8)–C(16)	115·4 (1·1)				

Three further cycles with isotropic temperature factors lowered R to 0·137. The three subsequent cycles with anisotropic temperature coefficients gave $R = 0{\cdot}100$. At this stage the positions of the H atoms of the benzene rings were calculated from geometrical considerations (by using the program *BONDAT* of the XRAY system), whereas the H atoms of the two methyl groups were located on a difference Fourier map. They were then 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 0·082. A systematic weight analysis was then carried out which led to the following weighting scheme: $w = (|F_o|/29)^2$ for $|F_o| < 29$ and $w = (29/|F_o|)^2$ for $|F_o| > 29$. However, the value of R remained the same in the two subsequent cycles ($R = 0{\cdot}082$, $R_w = 0{\cdot}068$). The average shift/error ratio of the last cycle was less than 0·4. The final difference Fourier map was virtually featureless.

The final positional parameters and equivalent isotropic temperature factors for the non-hydrogen atoms are given in Table 2. The final coordinates and isotropic temperature factors for the H atoms are shown in Table 3. Interatomic distances and bond angles are given in Tables 4 and 5.*

Description of the structure and discussion

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

To the two central five-membered isoxazoline (*P*1) and cyclopentenone (*P*2) rings, at an angle of 110·6° with each other, which compares very well with the angle 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), are linked three benzene rings and two methoxycarbonyl groups (*K*1,*K*2) with different orientations. The benzene rings (*B*1,*B*2,*B*3) and the methoxycarbonyl groups (H atoms excepted) are planar to a good approximation, whereas the five-membered rings deviate considerably from ideal planarity. In Table 6 are listed the least-squares planes through *P*1 and *P*2, together with the dihedral angles between them and the planes of the benzene rings.

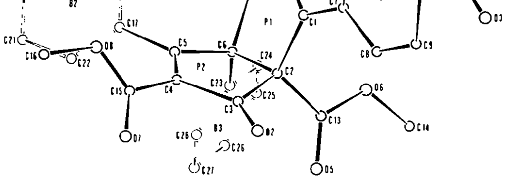


Fig. 1. Projection of a NPCCI molecule parallel to [010].

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* Lists of structure factors, anisotropic thermal parameters for the non-hydrogen atoms, and tables of C—H bond distances, C—C—H bond angles and least-squares planes through the benzene rings have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33267 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

As in CPCI mentioned above, the characteristic feature of the molecule is the coplanar system, formed by the isoxazoline ring *P*1 and the benzene ring *B*1 ($B1 \wedge P1 = 22{\cdot}9^\circ$) due to the conjugation between them. In this case, however, the system is only roughly coplanar, whereas in CPCI it is almost ideal ($B1 \wedge P1 = 6{\cdot}3^\circ$). The plane of the nitro group (*N*) is virtually a continuation of the ring *B*1, the angle between them being only 4·5°. Finally, the benzene ring *B*1 is perpendicular to the ring *P*2 and the group *K*2, and almost perpendicular to the group *K*1, the corresponding angles being 89·4, 89·2 and 81·9° respectively. Also, the ring *P*1 is almost normal to *K*1 and *K*2 (83·2 and 82·2° respectively). *B*1 and *K*1 are in *cis* positions as regards the isoxazoline ring.

Table 6. *Least-squares planes in NPCCI, with displacements of atoms from the planes* (Å)

The equation for a plane is in the form $AX + BY + CZ = D$ and refers to orthogonal axes. The coordinates *X*, *Y* and *Z* are expressed in Å; *D* is the distance of the plane from the origin. 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 *P*1: five-membered isoxazoline ring

$$0{\cdot}74125X - 0{\cdot}64209Y + 0{\cdot}19561Z = 7{\cdot}01809$$

O(1)	−0·09 (8)	C(7)*	0·12
N(1)	0·03		
C(1)	0·03		
C(2)	−0·07		
C(6)	0·10		

Plane *P*2: five-membered cyclopentenone ring

$$-0{\cdot}14811X - 0{\cdot}45050Y + 0{\cdot}88040Z = 4{\cdot}69406$$

C(2)	0·11 (9)	O(2)*	−0·21
C(3)	−0·09	C(15)*	0·12
C(4)	0·03	C(17)*	0·04
C(5)	0·04		
C(6)	−0·09		

Plane *K*1: methoxycarbonyl group 1 [attached to C(2)]

$$0{\cdot}55583X + 0{\cdot}83119Y + 0{\cdot}01327Z = 5{\cdot}55389$$

O(5)	−0·001 (2)	C(2)*	0·113
O(6)	−0·002	H1(C14)*	0·863
C(13)	0·002	H2(C14)*	0·057
C(14)	0·001	H3(C14)*	−0·629

Plane *K*2: methoxycarbonyl group 2 [attached to C(4)]

$$0{\cdot}55967X + 0{\cdot}75178Y - 0{\cdot}34872Z = 3{\cdot}49311$$

O(7)	−0·004 (6)	C(4)*	0·106
O(8)	−0·006	H1(C16)*	−0·830
C(15)	0·007	H2(C16)*	0·710
C(16)	0·003	H3(C16)*	0·139

Plane *N*: nitro group [attached to C(10)]

$$0{\cdot}76036X - 0{\cdot}63618Y - 0{\cdot}13089Z = 4{\cdot}29600$$

O(3)	0·003 (6)	N(2)	−0·008
O(4)	0·003	C(10)	0·002

Dihedral angles between planes (°) (*B*1, *B*2 and *B*3 are the benzene rings)

$B1 \wedge B2$ 40·6	$B2 \wedge B3$ 74·0	$B3 \wedge P1$ 58·5	$P1 \wedge P2$ 69·4
$B1 \wedge B3$ 72·5	$B2 \wedge P1$ 25·6	$B3 \wedge P2$ 77·4	$P1 \wedge K1$ 83·2
$B1 \wedge P1$ 22·9	$B2 \wedge P2$ 49·4	$B3 \wedge K1$ 46·8	$P1 \wedge K2$ 82·2
$B1 \wedge P2$ 89·4	$B2 \wedge K1$ 61·4	$B3 \wedge K2$ 61·6	$P1 \wedge N$ 18·8
$B1 \wedge K1$ 81·9	$B2 \wedge K2$ 56·9	$B3 \wedge N$ 68·4	
$B1 \wedge K2$ 89·2	$B2 \wedge N$ 38·5		
$B1 \wedge N$ 4·5			
$P2 \wedge K1$ 63·6	$K1 \wedge K2$ 21·4	$K2 \wedge N$ 89·6	
$P2 \wedge K2$ 43·2	$K1 \wedge N$ 83·8		
$P2 \wedge N$ 86·6			

The carbonyl oxygen atom O(2) occupies a prominent position in the molecule, as it does in the CPCI molecule: it lies at almost equal distances from the atoms C(8), O(5) and O(7), belonging to the benzene ring *B*1 and the methoxycarbonyl groups *K*1 and *K*2 respectively [O(2)–C(8) = 3·25 (2), O(2)–O(5) = 3·00 (1) and O(2)–O(7) = 3·04 (1) Å]. It occupies the apex of a flat trigonal pyramid, whose base is formed by these atoms.

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 and *B*3 are 1·39 (2), 1·37 (3) and 1·39 (2) Å respectively. However, the ring *B*2 shows some deviations: three bond lengths are rather short (1·31–1·34 Å) with corresponding lengthenings of two other bonds (1·41–1·44 Å). Characteristically, the temperature coefficients of the C atoms in this ring are much higher than those in the other benzene rings (see Table 2).

The N–O distances in the NO_2 groups are normal [1·19 (2) and 1·25 (2) Å respectively], but, as in the majority of cases found in the literature, they differ

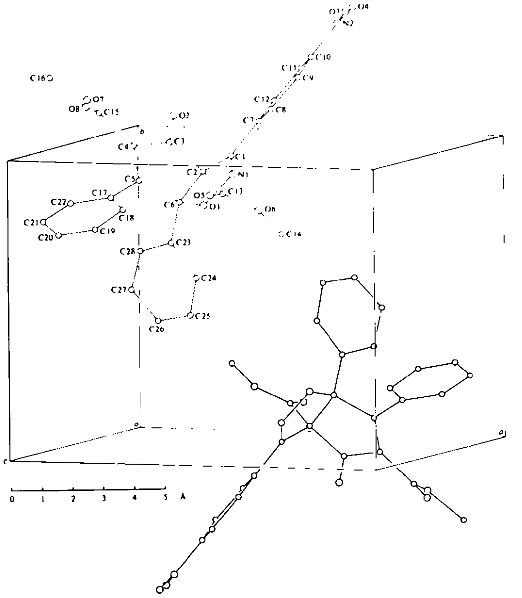

Fig. 2. Clinographic projection of the unit cell, showing the molecular packing.

Table 7. *Intermolecular distances* (Å) *in NPCCI*

O(2)···H3(C14)^1	2·92 (10)
O(4)···H(C24)^1	2·48 (9)
O(5)···H(C9)^1	2·45 (9)
O(7)···H(C22)^1	2·76 (12)
O(7)···H(C28)^2	2·52 (8)

Symmetry code

(1) $1-x, 1-y, 1-z$
(2) $2-x, 1-y, 1-z$

slightly. The bond length C_{ar}—N(2) = 1·51 (2) Å is longer than the mean value of 1·463 (2) Å deduced by Domenicano, Vaciago & Coulson (1975) for a number of *p*-substituted nitrobenzenes, but within the range of values reported for compounds with NO_2 groups (*cf.* Kokkou & Rentzeperis, 1975).

The general features of the five-membered isoxazoline and cyclopentenone rings are normal. Of the two methoxycarbonyl groups, *K*1 [connected to C(2)] shows normal bond lengths, whereas in *K*2 [connected to C(4)] the distances are shortened due to the formation of the conjugated system O(7)=C(15)—C(4)=C(5).

The intramolecular contact distances O(2)—H(C8) = 2·77 (10) Å, O(2)—O(5) = 3·00 (1) Å and O(2)—O(7) = 3·04 (1) Å almost coincide with the sum of the van der Waals radii of the corresponding atoms (Bondi, 1964). On the other hand, the contact distances of the two O atoms of the nitro group with neighbouring H atoms, *i.e.* O(3)—H(C9) = 2·38 (10) Å and O(4)—H(C11) = 2·59 (9) Å, are shorter than the sum of the van der Waals radii. All other contact distances are within the expected values.

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