

C3—C4	1.385 (3)	C9—C10	1.353 (3)
C4—O3	1.364 (3)	C10—C11	1.371 (3)
C4—C5	1.376 (3)	C11—N2	1.372 (3)
C5—C6	1.387 (3)	C11—C12	1.469 (3)
O1—N1	1.225 (3)	O4—N2	1.324 (2)
C6—C1—C2	122.6 (2)	O1—N1—C1	118.3 (2)
C6—C1—N1	118.3 (2)	N2—C7—C8	121.5 (2)
C2—C1—N1	119.2 (2)	C9—C8—C7	118.9 (3)
C3—C2—C1	119.0 (2)	C10—C9—C8	118.6 (2)
C2—C3—C4	119.5 (2)	C11—C10—C9	122.2 (2)
O3—C4—C5	121.7 (2)	N2—C11—C10	117.7 (2)
O3—C4—C3	117.5 (2)	N2—C11—C12	116.6 (2)
C5—C4—C3	120.7 (2)	C10—C11—C12	125.7 (2)
C6—C5—C4	119.8 (2)	O4—N2—C7	120.4 (2)
C1—C6—C5	118.4 (2)	O4—N2—C11	118.6 (2)
O2—N1—O1	124.1 (2)	C7—N2—C11	121.0 (2)
O2—N1—C1	117.5 (3)		

The ring H atoms were included at geometrically idealized positions with the exception of atom HO3 which was located from a Fourier difference map and its coordinates refined. An isotropic displacement parameter of  $6.0 \text{ \AA}^2$  was assigned to all H atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *VAXSDP* (Frenz, 1978). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *VAXSDP*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1163). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## A Multichromophoric Bicyclo[2.2.2]-octenone and a Photorearranged Ester Derivative

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## Abstract

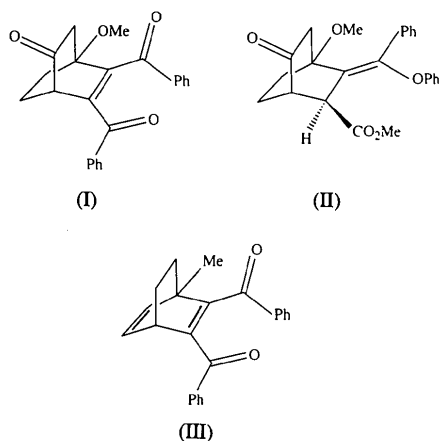
Both the title compound, 5,6-dibenzoyl-4-methoxybicyclo[2.2.2]oct-5-en-2-one,  $C_{23}H_{20}O_4$ , and its photolysis product obtained in the solution phase, 4-methoxy-6-*exo*-methoxycarbonyl-5-(1-phenoxy-1-phenylmethylene)bicyclo[2.2.2]octan-2-one<sup>†</sup>,  $C_{24}H_{24}O_5$ , both crystallize in the monoclinic space group  $P2_1/c$ . The structures were solved by direct methods. The conformations established for the two compounds facilitate understanding of the mechanism of the photoreaction in solution.

## Comment

UV irradiation of a solution of 5,6-dibenzoyl-4-methoxybicyclo[2.2.2]oct-5-en-2-one, (I), in methanol gave the ester 4-methoxy-6-*exo*-methoxycarbonyl-5-(1-phenoxy-1-phenylmethylene)bicyclo[2.2.2]octan-2-one, (II), via a ketene intermediate formed by a 1,5-phenyl migration in compound (I) (Lahiri & Singh, 1989). The <sup>1</sup>H NMR spectrum of compound (II) showed a highly shielded  $\delta$  value (2.55 p.p.m.) for the methoxy bridge-

<sup>†</sup> IUPAC name: methyl 4-methoxy-2-oxo-5-(1-phenoxy-1-phenylmethylene)bicyclo[2.2.2]octanyl-6-*exo*-carboxylate.

head proton, instead of a value in the normal range  $\delta = 3\text{--}4$  p.p.m. This effect is possible only if the methoxy group is shielded by the adjacent phenyl-ring current. Interestingly, in the unsymmetrical *cis*-1,2-dibenzoyl-alkene system, it has been observed (Singh, Sinha & Lahiri, 1992) that the 1,5-phenyl migration takes place from the benzoyl group attached to the more crowded olefinic C atom, for example, atom C5 in compound (III). The formation of compound (II), therefore, must have occurred either *via* a different migratory route or *via* a bond rotation of one of the benzoyl groups, which is likely to be a facile process in solution. To decide upon the reaction pathway for this phototransformation, the molecular structures of both compounds (I) and (II) had to be established unambiguously by X-ray crystallography.



*ORTEP*II (Johnson, 1976) plots for the parent and product molecules with the atom-labelling schemes are shown in Figs. 1 and 2, respectively. In the parent compound, (I), the benzoyl carbonyl groups C10=O3 and C17=O4 are *anti* and *syn*, respectively, in relation to the C5=C6 double bond, a situation similar to that observed in compound (III) (Mukhopadhyay, Ray & Lahiri, 1987). But Fig. 2 reveals that 1,5-phenyl migration during phototransformation has taken place from the 6-benzoyl moiety and not from the 5-benzoyl moiety as in the case of compound (III). The *syn*-phenoxy conformation of the resulting ester, (II), explains the observed highly shielded  $\delta$  value of the methoxy bridgehead group due to the adjacent phenyl ring C11–C16 (Fig. 2). This clearly suggests that during the photoreaction in methanol solution, the molecule of (I) flips to its 5-*syn*,6-*anti* conformer, thus facilitating migration of the phenyl group from C17 to O3, resulting in a ketene which on reaction with methanol produces the ester (II). This process, involving a flipping, is evidently very unlikely to occur in a crystal and quite expectedly, photolysis in the solid state has been observed to yield the other 1,5-phenyl migration isomer (Maji & Lahiri, unpublished).

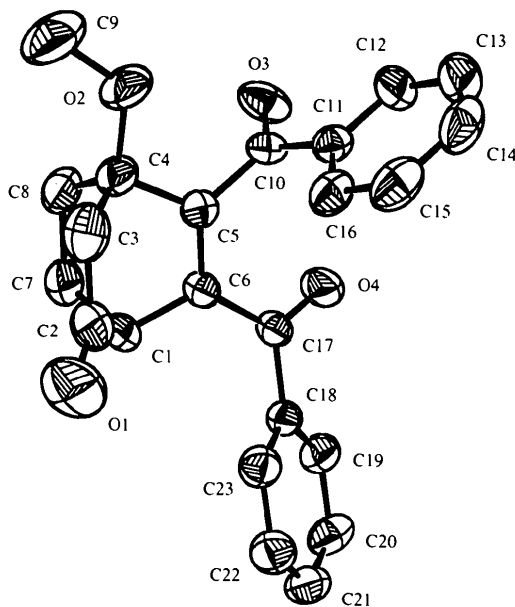


Fig. 1. *ORTEP*II (Johnson, 1976) diagram of compound (I) with displacement ellipsoids drawn at the 50% probability level.

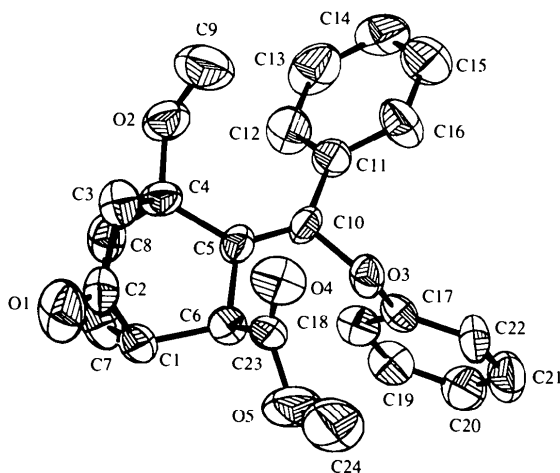


Fig. 2. *ORTEP*II (Johnson, 1976) diagram of compound (II) with displacement ellipsoids drawn at the 50% probability level.

## Experimental

The enone (I) was prepared by the Diels–Alder addition of 1,3-dimethoxy-1,3-cyclohexadiene and dibenzoylacetylene, followed by hydrolysis of the cycloadduct. Ester (II) was obtained from compound (I) by UV irradiation. The densities  $D_m$  of both compounds were measured by flotation in aqueous KBr.

### Compound (I)

#### Crystal data

C<sub>23</sub>H<sub>20</sub>O<sub>4</sub>  
 $M_r = 360.41$

Mo  $K\alpha$  radiation  
 $\lambda = 0.7107 \text{ \AA}$

Monoclinic	Cell parameters from 25 reflections
$P2_1/c$	$\theta = 5.5\text{--}12.5^\circ$
$a = 11.059$ (4) Å	$\mu = 0.089$ mm <sup>-1</sup>
$b = 14.188$ (5) Å	$T = 300$ K
$c = 11.996$ (5) Å	Prismatic
$\beta = 102.39$ (3)°	$0.54 \times 0.38 \times 0.36$ mm
$V = 1838.4$ (12) Å <sup>3</sup>	Colourless
$Z = 4$	
$D_x = 1.30$ Mg m <sup>-3</sup>	
$D_m = 1.29$ Mg m <sup>-3</sup>	

**Data collection**

Nicolet R3m/V diffractometer	$\theta_{\max} = 25^\circ$
$\omega/2\theta$ scans	$h = 0 \rightarrow 14$
Absorption correction: none	$k = 0 \rightarrow 17$
3012 measured reflections	$l = -15 \rightarrow 15$
2853 independent reflections	2 standard reflections monitored every 100 reflections
1796 observed reflections	intensity decay: no significant variation
$[I > 2.5\sigma(I)]$	
$R_{\text{int}} = 0.0135$	

**Refinement**

Refinement on $F$	$(\Delta/\sigma)_{\max} = 0.208$
$R = 0.047$	$\Delta\rho_{\max} = 0.27$ e Å <sup>-3</sup>
$wR = 0.052$	$\Delta\rho_{\min} = -0.20$ e Å <sup>-3</sup>
$S = 1.169$	Extinction correction: none
1796 reflections	Atomic scattering factors from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
220 parameters	
H atoms fixed	
$w = 1.1685/[\sigma^2(F) + 0.000625F^2]$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>) for (I)

	$x$	$y$	$z$	$U_{\text{eq}}$
C1	0.0146 (3)	0.1062 (2)	0.7692 (3)	0.043
C2	0.0442 (3)	0.1048 (3)	0.8975 (3)	0.060
C3	0.1808 (3)	0.0965 (3)	0.9464 (3)	0.061
C4	0.2496 (3)	0.0882 (2)	0.8488 (3)	0.042
C5	0.1912 (3)	0.0080 (2)	0.7732 (2)	0.034
C6	0.0692 (3)	0.0168 (2)	0.7324 (2)	0.034
C7	0.0851 (3)	0.1901 (2)	0.7315 (3)	0.054
C8	0.2255 (3)	0.1786 (2)	0.7767 (3)	0.050
C9	0.4536 (4)	0.1317 (3)	0.9529 (4)	0.097
C10	0.2729 (2)	-0.0675 (2)	0.7427 (2)	0.037
C11	0.2794 (3)	-0.1601 (2)	0.8002 (3)	0.037
C12	0.3528 (3)	-0.2302 (2)	0.7687 (3)	0.050
C13	0.3648 (3)	-0.3157 (3)	0.8235 (3)	0.061
C14	0.3059 (4)	-0.3314 (3)	0.9122 (3)	0.059
C15	0.2323 (3)	-0.2633 (3)	0.9432 (3)	0.058
C16	0.2182 (3)	-0.1770 (2)	0.8876 (3)	0.045
C17	-0.0026 (3)	-0.0539 (2)	0.6533 (2)	0.036
C18	-0.1375 (3)	-0.0643 (2)	0.6466 (2)	0.034
C19	-0.2124 (3)	-0.0859 (2)	0.5416 (3)	0.045
C20	-0.3387 (3)	-0.0970 (2)	0.5318 (3)	0.054
C21	-0.3902 (3)	-0.0880 (2)	0.6252 (3)	0.056
C22	-0.3154 (3)	-0.0687 (2)	0.7298 (3)	0.060
C23	-0.1892 (3)	-0.0558 (2)	0.7406 (3)	0.048
O1	-0.0315 (3)	0.1087 (2)	0.9565 (2)	0.101
O2	0.3768 (2)	0.0655 (2)	0.8871 (2)	0.064
O3	0.3360 (2)	-0.0489 (2)	0.6745 (2)	0.062
O4	0.0494 (2)	-0.1016 (2)	0.5941 (2)	0.063

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

Table 2. Selected geometric parameters (Å, °) for (I)

C2—C1	1.503 (5)	C6—C5	1.339 (4)
C6—C1	1.511 (4)	C10—C5	1.496 (4)
C7—C1	1.543 (4)	C17—C6	1.488 (4)
C3—C2	1.504 (5)	C8—C7	1.539 (4)
O1—C2	1.207 (4)	O2—C9	1.393 (4)
C4—C3	1.531 (4)	C11—C10	1.478 (4)
C5—C4	1.511 (4)	O3—C10	1.213 (3)
C8—C4	1.538 (4)	C18—C17	1.484 (4)
O2—C4	1.420 (4)	O4—C17	1.212 (3)
C6—C1—C2	106.0 (3)	C5—C6—C1	114.0 (3)
C7—C1—C2	107.3 (3)	C17—C6—C1	124.0 (3)
C7—C1—C6	107.6 (2)	C17—C6—C5	121.9 (3)
C3—C2—C1	112.3 (3)	C8—C7—C1	110.5 (3)
O1—C2—C1	124.9 (3)	C7—C8—C4	109.1 (2)
O1—C2—C3	122.7 (3)	C11—C10—C5	119.6 (3)
C4—C3—C2	109.3 (3)	O3—C10—C5	118.3 (3)
C5—C4—C3	107.4 (3)	O3—C10—C11	122.0 (3)
C8—C4—C3	108.1 (3)	C12—C11—C10	119.0 (3)
C8—C4—C5	106.8 (2)	C16—C11—C10	121.6 (3)
O2—C4—C3	113.0 (3)	C18—C17—C6	119.6 (3)
O2—C4—C5	106.8 (2)	O4—C17—C6	119.6 (3)
O2—C4—C8	114.3 (3)	O4—C17—C18	120.8 (3)
C6—C5—C4	114.6 (3)	C19—C18—C17	118.0 (3)
C10—C5—C4	118.9 (2)	C23—C18—C17	122.4 (3)
C10—C5—C6	126.2 (3)	C9—O2—C4	118.1 (3)

Table 3. Hydrogen-bonding geometry (Å, °) for (I)

D—H...A	H...A	D...A	D—H...A
C16—H16...O1 <sup>i</sup>	2.434 (13)	3.218 (5)	138.8 (11)
C21—H21...O3 <sup>ii</sup>	2.393 (13)	3.255 (6)	149.2 (10)

Symmetry codes: (i)  $-x, -y, 2-z$ ; (ii)  $x-1, y, z$ .**Compound (II)****Crystal data**

$\text{C}_{24}\text{H}_{24}\text{O}_5$
$M_r = 392.46$
Monoclinic
$P2_1/c$
$a = 6.630$ (3) Å
$b = 21.780$ (6) Å
$c = 14.759$ (5) Å
$\beta = 98.35$ (3)°
$V = 2108.7$ (13) Å <sup>3</sup>
$Z = 4$
$D_x = 1.24$ Mg m <sup>-3</sup>
$D_m = 1.25$ Mg m <sup>-3</sup>

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 10\text{--}30^\circ$
$\mu = 0.62$ mm <sup>-1</sup>
$T = 300$ K
Prismatic
$0.420 \times 0.160 \times 0.075$ mm
Colourless

**Data collection**

Locally assembled computer-controlled diffractometer system incorporating Huber crystal orienter	$R_{\text{int}} = 0.0132$
$\omega/2\theta$ scans	$\theta_{\max} = 55^\circ$
Absorption correction: none	$h = -6 \rightarrow 6$
2359 measured reflections	$k = 0 \rightarrow 21$
1745 independent reflections	$l = 0 \rightarrow 14$
1323 observed reflections	3 standard reflections monitored every 50 reflections
$[I > 2\sigma(I)]$	intensity decay: no significant variation

**Refinement**

Refinement on $F$	$(\Delta/\sigma)_{\max} = 0.160$
$R = 0.063$	$\Delta\rho_{\max} = 0.19$ e Å <sup>-3</sup>
$wR = 0.064$	$\Delta\rho_{\min} = -0.29$ e Å <sup>-3</sup>
$S = 1.274$	Extinction correction: none

1323 reflections

238 parameters

H atom fixed

$$w = 1.2738/[\sigma^2(F) + 0.00005F^2]$$

Atomic scattering factors  
from *International Tables*  
for *X-ray Crystallography*  
(1974, Vol. IV)

C16—H16···O5 <sup>iii</sup>	2.717 (18)	3.450 (8)	124.9 (8)
C18—H18···O4 <sup>ii</sup>	2.511 (15)	3.252 (8)	124.9 (13)
C20—H20···O1 <sup>i</sup>	2.633 (18)	3.380 (6)	125.8 (6)

Symmetry codes: (i)  $1-x, -y, 2-z$ ; (ii)  $x-1, y, z$ ; (iii)  $x, \frac{1}{2}-y, z-\frac{1}{2}$ ; (iv)  $x-1, \frac{1}{2}-y, z-\frac{1}{2}$ .

Table 4. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (II)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{eq}$
C1	0.3735 (12)	0.1088 (3)	0.9210 (5)	0.049
C2	0.5405 (13)	0.0619 (3)	0.9232 (5)	0.053
C3	0.5420 (10)	0.0309 (3)	0.8303 (4)	0.048
C4	0.3474 (12)	0.0517 (3)	0.7638 (5)	0.046
C5	0.3470 (11)	0.1206 (3)	0.7506 (5)	0.043
C6	0.3921 (12)	0.1546 (3)	0.8422 (4)	0.040
C7	0.1684 (11)	0.0755 (4)	0.8978 (5)	0.080
C8	0.1596 (12)	0.0356 (3)	0.8107 (5)	0.057
C9	0.4984 (14)	0.0154 (4)	0.6348 (5)	0.090
C10	0.3057 (12)	0.1532 (3)	0.6751 (5)	0.048
C11	0.2334 (9)	0.1364 (2)	0.5777 (3)	0.048
C12	0.0734 (9)	0.0951 (2)	0.5546 (3)	0.069
C13	0.0088 (9)	0.0805 (2)	0.4630 (3)	0.083
C14	0.1042 (9)	0.1071 (2)	0.3945 (3)	0.079
C15	0.2642 (9)	0.1484 (2)	0.4176 (3)	0.097
C16	0.3288 (9)	0.1631 (2)	0.5092 (3)	0.070
C17	0.1830 (8)	0.2571 (2)	0.6693 (3)	0.041
C18	-0.0145 (8)	0.2393 (2)	0.6794 (3)	0.049
C19	-0.1737 (8)	0.2817 (2)	0.6647 (3)	0.058
C20	-0.1353 (8)	0.3419 (2)	0.6399 (3)	0.070
C21	0.0623 (8)	0.3597 (2)	0.6297 (3)	0.073
C22	0.2214 (8)	0.3173 (2)	0.6444 (3)	0.055
C23	0.6086 (16)	0.1822 (4)	0.8539 (5)	0.049
C24	0.8101 (15)	0.2626 (4)	0.9249 (6)	0.107
O1	0.6691 (9)	0.0494 (2)	0.9887 (3)	0.077
O2	0.3255 (9)	0.0161 (2)	0.6824 (4)	0.063
O3	0.3456 (7)	0.2168 (2)	0.6857 (3)	0.046
O4	0.7491 (9)	0.1630 (3)	0.8216 (4)	0.068
O5	0.6119 (10)	0.2318 (3)	0.9062 (4)	0.083

For both compounds, full-matrix anisotropic refinement on  $F$  of the non-H atoms was carried out using *SHELX76* (Sheldrick, 1976). H atoms were placed at calculated positions, with a fixed common  $U$  of  $0.08 \text{\AA}^2$ , and were allowed to ride on their corresponding C atoms. During the final stages of refinement on compound (II), the C atoms of the benzene rings were constrained to a hexagonal geometry. Weights used were of the form  $w = k[\sigma^2(F) + gF^2]$ , with  $g = 0.000625$  for (I) and  $0.00005$  for (II). With an initial value of  $k = 1$ , the final recalculated values obtained were  $k = 1.1685$  and  $1.2738$  for (I) and (II), respectively. All calculations were carried out using a VAX3400 computer at the Computer Centre, Indian Association for the Cultivation of Science, Calcutta, India.

Data collection: *SHELXTL-Plus88* (Sheldrick, 1988) for (I); local program for (II). Cell refinement: *SHELXTL-Plus88* for (I); local program for (II). Data reduction: *SHELXTL-Plus88* for (I); local program for (II). For both compounds, program(s) used to solve structures: *MULTAN88* (Debaerdemaeker *et al.*, 1988); molecular graphics: *ORTEPII* (Johnson, 1976). Calculation of geometric parameters: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DE1017). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 5. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (II)

C2—O1	1.223 (9)	C7—C1	1.535 (9)
C4—O2	1.419 (8)	C3—C2	1.530 (9)
C9—O2	1.430 (10)	C4—C3	1.570 (9)
C10—O3	1.415 (8)	C5—C4	1.514 (9)
C17—O3	1.384 (6)	C8—C4	1.550 (10)
C23—O4	1.183 (10)	C6—C5	1.533 (9)
C23—O5	1.325 (10)	C10—C5	1.317 (10)
C24—O5	1.466 (10)	C23—C6	1.543 (12)
C2—C1	1.503 (10)	C8—C7	1.547 (9)
C6—C1	1.551 (9)	C11—C10	1.493 (8)
C9—O2—C4	115.5 (7)	C10—C5—C4	129.8 (8)
C17—O3—C10	118.2 (6)	C10—C5—C6	118.5 (7)
C24—O5—C23	114.9 (8)	C5—C6—C1	108.9 (7)
C6—C1—C2	108.4 (7)	C23—C6—C1	109.9 (7)
C7—C1—C2	108.1 (7)	C23—C6—C5	110.3 (7)
C7—C1—C6	107.5 (7)	C8—C7—C1	112.1 (7)
C1—C2—O1	126.5 (9)	C7—C8—C4	108.0 (7)
C3—C2—O1	121.1 (8)	C5—C10—O3	114.9 (7)
C3—C2—C1	112.4 (8)	C11—C10—O3	112.1 (7)
C4—C3—C2	108.7 (7)	C11—C10—C5	132.9 (7)
C3—C4—O2	110.2 (7)	C12—C11—C10	121.3 (4)
C5—C4—O2	115.8 (7)	C16—C11—C10	118.7 (4)
C5—C4—C3	110.6 (7)	C18—C17—O3	121.6 (4)
C8—C4—O2	105.3 (7)	C22—C17—O3	118.4 (4)
C8—C4—C3	107.3 (7)	O5—C23—O4	125.2 (11)
C8—C4—C5	107.2 (7)	C6—C23—O4	126.2 (10)
C6—C5—C4	111.7 (7)	C6—C23—O5	108.6 (9)

Table 6. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ) for (II)

D—H···A	H···A	D···A	D—H···A
C7—H7A···O1 <sup>i</sup>	2.508 (19)	3.293 (9)	128.7 (8)
C7—H7B···O4 <sup>ii</sup>	2.382 (14)	3.423 (10)	161.5 (14)