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(E)-Ethyl 2-benzoyl-4-(naphthalen-2-yl)-4-oxobut-2-enoate

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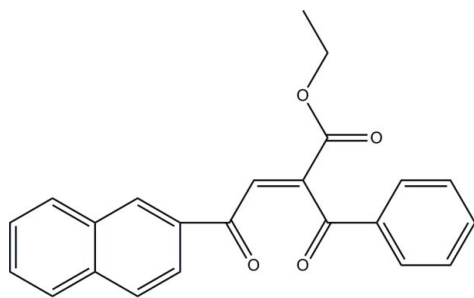
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.054; wR factor = 0.149; data-to-parameter ratio = 13.7.

The title compound, $\text{C}_{23}\text{H}_{18}\text{O}_4$, is a 1,4-enedione compound which contains a naphthalene ring and a benzene ring. The dihedral angle between the ring systems is $74.9(2)^\circ$. In the crystal, the molecules form π - π stacking interactions between naphthalene rings of inversion-related molecules, with an interplanar spacing of $3.499(2)$ Å.

Related literature

For the preparation of the title compound, see: Gao *et al.* (2010). For related structures, see: Prakash *et al.* (2005); Raj *et al.* (1996).



Experimental

Crystal data

$\text{C}_{23}\text{H}_{18}\text{O}_4$	$\gamma = 110.648(3)^\circ$
$M_r = 358.37$	$V = 912.6(3)$ Å ³
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.8571(13)$ Å	Mo $K\alpha$ radiation
$b = 9.6157(16)$ Å	$\mu = 0.09$ mm ⁻¹
$c = 13.934(2)$ Å	$T = 298$ K
$\alpha = 99.364(3)^\circ$	$0.16 \times 0.12 \times 0.10$ mm
$\beta = 105.094(3)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	3355 independent reflections
5661 measured reflections	2818 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.065$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$	245 parameters
$wR(F^2) = 0.149$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.18$ e Å ⁻³
3355 reflections	$\Delta\rho_{\text{min}} = -0.19$ e Å ⁻³

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*, *SHELXL97* and *publCIF* (Westrip, 2010).

The author are grateful to Dr Xiang-Gao Meng for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2318).

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supporting information

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(E)-Ethyl 2-benzoyl-4-(naphthalen-2-yl)-4-oxobut-2-enoate**Liuming Wu, Cong Deng and Yan Yang****S1. Comment**

The 1,4-enedione framework is frequently found in bioactive natural products and medicinal compounds. In addition, by virtue of their multifunctional composition, 1,4-enediones could serve as versatile precursors for heterocycle synthesis, Diels-Alder cycloaddition, as well as many other useful transformations. We report here the crystal structure of the title compound (Fig. 1). The crystal packing exhibits offset π - π stacking interactions.

S2. Experimental

The title compound was synthesized according to the reported literature (Gao *et al.*, 2010). Crystals suitable for X-ray diffraction were grown by slow evaporation of a ethyl acetate-hexane (2:1) solution of the title compound at 293 K.

S3. Refinement

All H atoms were positioned in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{Me}})$.

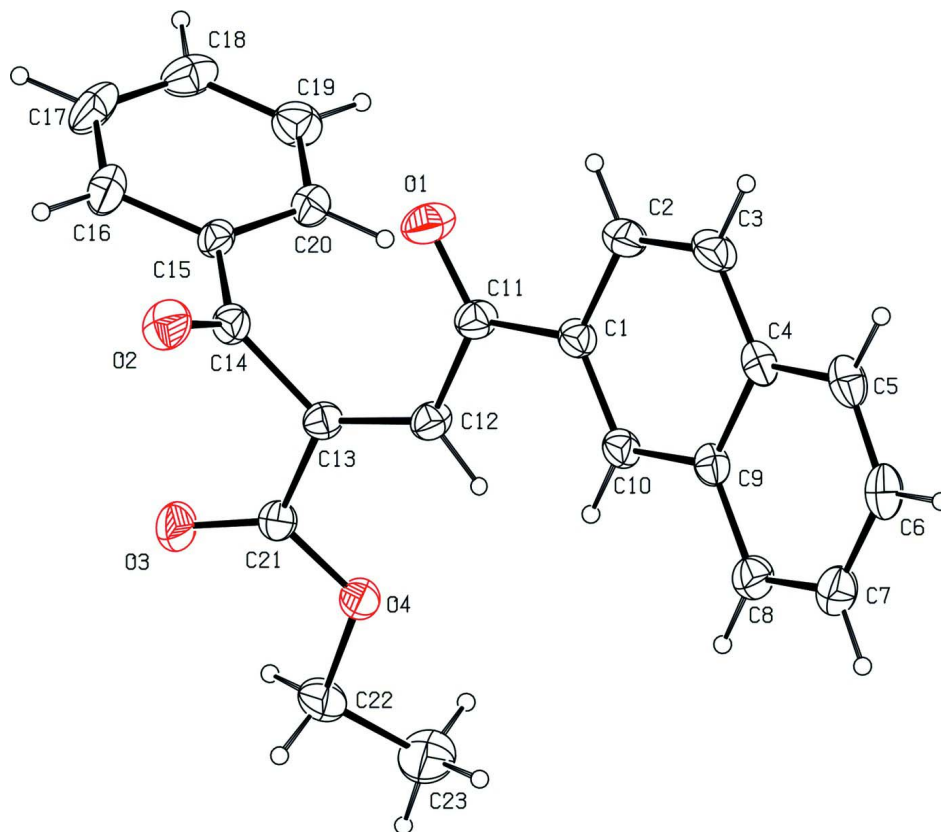


Figure 1

A view of the compound with displacement ellipsoids drawn at the 30% probability level.

(E)-Ethyl 2-benzoyl-4-(naphthalen-2-yl)-4-oxobut-2-enoate

Crystal data

$C_{23}H_{18}O_4$

$M_r = 358.37$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 7.8571$ (13) Å

$b = 9.6157$ (16) Å

$c = 13.934$ (2) Å

$\alpha = 99.364$ (3)°

$\beta = 105.094$ (3)°

$\gamma = 110.648$ (3)°

$V = 912.6$ (3) Å³

$Z = 2$

$F(000) = 376$

$D_x = 1.304$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2846 reflections

$\theta = 2.4$ – 27.7 °

$\mu = 0.09$ mm⁻¹

$T = 298$ K

Block, colorless

$0.16 \times 0.12 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

5661 measured reflections

3355 independent reflections

2818 reflections with $I > 2\sigma(I)$

$R_{int} = 0.065$

$\theta_{max} = 25.5$ °, $\theta_{min} = 2.4$ °

$h = -8 \rightarrow 9$

$k = -11 \rightarrow 9$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.149$

$S = 1.05$

3355 reflections

245 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.1681P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.18 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.7525 (2)	0.3380 (2)	0.02603 (13)	0.0480 (4)
C2	0.6361 (3)	0.2384 (2)	-0.07533 (14)	0.0594 (5)
H2	0.6065	0.1330	-0.0892	0.071*
C3	0.5681 (3)	0.2958 (2)	-0.15181 (14)	0.0634 (5)
H3	0.4969	0.2295	-0.2183	0.076*
C4	0.6024 (3)	0.4539 (2)	-0.13326 (13)	0.0526 (4)
C5	0.5236 (3)	0.5168 (3)	-0.20967 (15)	0.0644 (5)
H5	0.4507	0.4533	-0.2768	0.077*
C6	0.5524 (3)	0.6673 (3)	-0.18670 (17)	0.0666 (6)
H6	0.4974	0.7056	-0.2377	0.080*
C7	0.6642 (3)	0.7654 (3)	-0.08709 (17)	0.0673 (6)
H7	0.6839	0.8689	-0.0721	0.081*
C8	0.7445 (3)	0.7107 (2)	-0.01181 (15)	0.0602 (5)
H8	0.8192	0.7776	0.0542	0.072*
C9	0.7167 (2)	0.5546 (2)	-0.03195 (13)	0.0479 (4)
C10	0.7908 (3)	0.4926 (2)	0.04557 (13)	0.0486 (4)
H10	0.8677	0.5582	0.1117	0.058*
C11	0.8160 (3)	0.2679 (2)	0.10791 (14)	0.0529 (4)
C12	0.9526 (3)	0.3702 (2)	0.21270 (14)	0.0508 (4)
H12	1.0266	0.4736	0.2201	0.061*
C13	0.9720 (2)	0.31813 (19)	0.29631 (13)	0.0458 (4)
C14	0.8554 (3)	0.1555 (2)	0.29805 (14)	0.0502 (4)
C15	0.9508 (2)	0.04721 (18)	0.29869 (13)	0.0466 (4)
C16	0.9100 (3)	-0.0649 (2)	0.35035 (17)	0.0670 (6)
H16	0.8236	-0.0716	0.3859	0.080*

C17	0.9978 (4)	-0.1679 (3)	0.3492 (2)	0.0811 (7)
H17	0.9701	-0.2430	0.3841	0.097*
C18	1.1244 (4)	-0.1585 (2)	0.29701 (19)	0.0737 (6)
H18	1.1812	-0.2285	0.2954	0.088*
C19	1.1676 (3)	-0.0474 (3)	0.24748 (18)	0.0707 (6)
H19	1.2550	-0.0409	0.2126	0.085*
C20	1.0828 (3)	0.0558 (2)	0.24846 (15)	0.0562 (5)
H20	1.1147	0.1322	0.2149	0.067*
C21	1.1107 (3)	0.4188 (2)	0.40120 (13)	0.0500 (4)
C22	1.3699 (3)	0.6602 (3)	0.49986 (16)	0.0761 (6)
H22A	1.4252	0.6045	0.5412	0.091*
H22B	1.3052	0.7056	0.5372	0.091*
C23	1.5227 (4)	0.7817 (3)	0.4819 (2)	0.0995 (9)
H23A	1.5909	0.7365	0.4485	0.149*
H23B	1.6114	0.8551	0.5470	0.149*
H23C	1.4664	0.8333	0.4386	0.149*
O1	0.7583 (2)	0.12815 (15)	0.09194 (11)	0.0762 (5)
O2	0.6987 (2)	0.12270 (17)	0.30777 (13)	0.0743 (4)
O3	1.1113 (3)	0.37690 (18)	0.47767 (11)	0.0809 (5)
O4	1.23194 (19)	0.55455 (14)	0.40043 (9)	0.0607 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0466 (9)	0.0505 (10)	0.0416 (9)	0.0165 (8)	0.0136 (7)	0.0100 (7)
C2	0.0688 (12)	0.0530 (11)	0.0468 (10)	0.0222 (9)	0.0148 (9)	0.0046 (8)
C3	0.0684 (13)	0.0687 (13)	0.0375 (10)	0.0227 (10)	0.0092 (9)	0.0027 (9)
C4	0.0498 (10)	0.0692 (12)	0.0398 (9)	0.0226 (9)	0.0194 (8)	0.0162 (8)
C5	0.0597 (12)	0.0911 (16)	0.0425 (10)	0.0295 (11)	0.0167 (9)	0.0247 (10)
C6	0.0641 (12)	0.0915 (16)	0.0609 (12)	0.0379 (12)	0.0272 (10)	0.0422 (12)
C7	0.0742 (14)	0.0692 (13)	0.0709 (14)	0.0330 (11)	0.0309 (11)	0.0340 (11)
C8	0.0648 (12)	0.0586 (11)	0.0517 (11)	0.0216 (9)	0.0159 (9)	0.0179 (9)
C9	0.0431 (9)	0.0567 (10)	0.0424 (9)	0.0166 (8)	0.0168 (7)	0.0160 (8)
C10	0.0474 (9)	0.0519 (10)	0.0365 (8)	0.0142 (8)	0.0098 (7)	0.0090 (7)
C11	0.0521 (10)	0.0458 (10)	0.0518 (10)	0.0169 (8)	0.0099 (8)	0.0115 (8)
C12	0.0506 (10)	0.0422 (9)	0.0497 (10)	0.0143 (8)	0.0088 (8)	0.0129 (8)
C13	0.0459 (9)	0.0436 (9)	0.0483 (9)	0.0203 (7)	0.0144 (7)	0.0130 (7)
C14	0.0471 (10)	0.0506 (10)	0.0478 (10)	0.0138 (8)	0.0173 (8)	0.0137 (8)
C15	0.0460 (9)	0.0398 (9)	0.0421 (9)	0.0088 (7)	0.0087 (7)	0.0126 (7)
C16	0.0611 (12)	0.0646 (12)	0.0654 (13)	0.0114 (10)	0.0177 (10)	0.0327 (10)
C17	0.0806 (16)	0.0546 (12)	0.0858 (16)	0.0134 (11)	0.0011 (13)	0.0409 (12)
C18	0.0717 (14)	0.0504 (12)	0.0797 (15)	0.0260 (10)	-0.0012 (12)	0.0122 (11)
C19	0.0788 (15)	0.0704 (14)	0.0694 (13)	0.0409 (12)	0.0239 (11)	0.0163 (11)
C20	0.0666 (12)	0.0527 (10)	0.0564 (11)	0.0274 (9)	0.0244 (9)	0.0224 (9)
C21	0.0552 (10)	0.0496 (10)	0.0468 (10)	0.0230 (8)	0.0165 (8)	0.0164 (8)
C22	0.0786 (15)	0.0692 (14)	0.0457 (11)	0.0092 (12)	0.0082 (10)	-0.0021 (10)
C23	0.0779 (17)	0.0856 (18)	0.0757 (16)	-0.0030 (14)	-0.0046 (13)	0.0066 (13)
O1	0.0878 (11)	0.0466 (8)	0.0652 (9)	0.0192 (7)	-0.0030 (8)	0.0097 (7)

O2	0.0568 (9)	0.0716 (9)	0.0978 (11)	0.0209 (7)	0.0394 (8)	0.0244 (8)
O3	0.1044 (12)	0.0704 (10)	0.0491 (8)	0.0171 (9)	0.0188 (8)	0.0249 (7)
O4	0.0654 (8)	0.0528 (8)	0.0406 (7)	0.0071 (6)	0.0084 (6)	0.0092 (6)

Geometric parameters (Å, °)

C1—C10	1.373 (2)	C13—C14	1.517 (2)
C1—C2	1.420 (3)	C14—O2	1.208 (2)
C1—C11	1.482 (2)	C14—C15	1.482 (3)
C2—C3	1.352 (3)	C15—C20	1.381 (3)
C2—H2	0.9300	C15—C16	1.383 (3)
C3—C4	1.413 (3)	C16—C17	1.392 (3)
C3—H3	0.9300	C16—H16	0.9300
C4—C5	1.419 (3)	C17—C18	1.365 (4)
C4—C9	1.421 (3)	C17—H17	0.9300
C5—C6	1.352 (3)	C18—C19	1.356 (3)
C5—H5	0.9300	C18—H18	0.9300
C6—C7	1.394 (3)	C19—C20	1.377 (3)
C6—H6	0.9300	C19—H19	0.9300
C7—C8	1.358 (3)	C20—H20	0.9300
C7—H7	0.9300	C21—O3	1.198 (2)
C8—C9	1.406 (3)	C21—O4	1.321 (2)
C8—H8	0.9300	C22—O4	1.453 (2)
C9—C10	1.410 (2)	C22—C23	1.459 (3)
C10—H10	0.9300	C22—H22A	0.9700
C11—O1	1.217 (2)	C22—H22B	0.9700
C11—C12	1.491 (2)	C23—H23A	0.9600
C12—C13	1.335 (2)	C23—H23B	0.9600
C12—H12	0.9300	C23—H23C	0.9600
C13—C21	1.492 (2)		
C10—C1—C2	118.91 (16)	C21—C13—C14	112.15 (14)
C10—C1—C11	122.81 (15)	O2—C14—C15	122.53 (16)
C2—C1—C11	118.14 (16)	O2—C14—C13	120.11 (16)
C3—C2—C1	120.47 (18)	C15—C14—C13	117.05 (15)
C3—C2—H2	119.8	C20—C15—C16	118.37 (18)
C1—C2—H2	119.8	C20—C15—C14	121.17 (15)
C2—C3—C4	121.70 (17)	C16—C15—C14	120.46 (17)
C2—C3—H3	119.2	C15—C16—C17	120.2 (2)
C4—C3—H3	119.2	C15—C16—H16	119.9
C3—C4—C5	123.50 (18)	C17—C16—H16	119.9
C3—C4—C9	118.46 (17)	C18—C17—C16	120.1 (2)
C5—C4—C9	118.00 (18)	C18—C17—H17	120.0
C6—C5—C4	121.28 (19)	C16—C17—H17	120.0
C6—C5—H5	119.4	C19—C18—C17	120.2 (2)
C4—C5—H5	119.4	C19—C18—H18	119.9
C5—C6—C7	120.40 (19)	C17—C18—H18	119.9
C5—C6—H6	119.8	C18—C19—C20	120.4 (2)

C7—C6—H6	119.8	C18—C19—H19	119.8
C8—C7—C6	120.4 (2)	C20—C19—H19	119.8
C8—C7—H7	119.8	C19—C20—C15	120.80 (18)
C6—C7—H7	119.8	C19—C20—H20	119.6
C7—C8—C9	121.17 (19)	C15—C20—H20	119.6
C7—C8—H8	119.4	O3—C21—O4	124.25 (17)
C9—C8—H8	119.4	O3—C21—C13	122.09 (17)
C8—C9—C10	122.55 (16)	O4—C21—C13	113.65 (15)
C8—C9—C4	118.77 (17)	O4—C22—C23	108.74 (19)
C10—C9—C4	118.61 (16)	O4—C22—H22A	109.9
C1—C10—C9	121.80 (16)	C23—C22—H22A	109.9
C1—C10—H10	119.1	O4—C22—H22B	109.9
C9—C10—H10	119.1	C23—C22—H22B	109.9
O1—C11—C1	121.54 (17)	H22A—C22—H22B	108.3
O1—C11—C12	118.93 (16)	C22—C23—H23A	109.5
C1—C11—C12	119.53 (15)	C22—C23—H23B	109.5
C13—C12—C11	122.11 (16)	H23A—C23—H23B	109.5
C13—C12—H12	118.9	C22—C23—H23C	109.5
C11—C12—H12	118.9	H23A—C23—H23C	109.5
C12—C13—C21	122.55 (16)	H23B—C23—H23C	109.5
C12—C13—C14	125.27 (16)	C21—O4—C22	116.98 (15)
C10—C1—C2—C3	1.5 (3)	C11—C12—C13—C21	178.92 (16)
C11—C1—C2—C3	177.41 (18)	C11—C12—C13—C14	-3.1 (3)
C1—C2—C3—C4	-2.8 (3)	C12—C13—C14—O2	-84.9 (2)
C2—C3—C4—C5	-175.90 (18)	C21—C13—C14—O2	93.3 (2)
C2—C3—C4—C9	2.0 (3)	C12—C13—C14—C15	101.4 (2)
C3—C4—C5—C6	176.54 (19)	C21—C13—C14—C15	-80.41 (19)
C9—C4—C5—C6	-1.4 (3)	O2—C14—C15—C20	153.77 (19)
C4—C5—C6—C7	1.2 (3)	C13—C14—C15—C20	-32.7 (2)
C5—C6—C7—C8	-0.4 (3)	O2—C14—C15—C16	-26.5 (3)
C6—C7—C8—C9	-0.3 (3)	C13—C14—C15—C16	147.00 (18)
C7—C8—C9—C10	-177.10 (18)	C20—C15—C16—C17	-1.3 (3)
C7—C8—C9—C4	0.1 (3)	C14—C15—C16—C17	178.96 (18)
C3—C4—C9—C8	-177.29 (18)	C15—C16—C17—C18	-0.1 (3)
C5—C4—C9—C8	0.7 (3)	C16—C17—C18—C19	1.1 (4)
C3—C4—C9—C10	0.0 (2)	C17—C18—C19—C20	-0.7 (3)
C5—C4—C9—C10	178.01 (16)	C18—C19—C20—C15	-0.8 (3)
C2—C1—C10—C9	0.4 (3)	C16—C15—C20—C19	1.7 (3)
C11—C1—C10—C9	-175.22 (16)	C14—C15—C20—C19	-178.54 (18)
C8—C9—C10—C1	176.00 (17)	C12—C13—C21—O3	170.97 (19)
C4—C9—C10—C1	-1.2 (3)	C14—C13—C21—O3	-7.2 (2)
C10—C1—C11—O1	169.80 (19)	C12—C13—C21—O4	-10.2 (2)
C2—C1—C11—O1	-5.9 (3)	C14—C13—C21—O4	171.62 (15)
C10—C1—C11—C12	-9.7 (3)	O3—C21—O4—C22	-2.0 (3)
C2—C1—C11—C12	174.56 (16)	C13—C21—O4—C22	179.16 (16)
O1—C11—C12—C13	-19.2 (3)	C23—C22—O4—C21	164.2 (2)
C1—C11—C12—C13	160.31 (17)		
