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2-Oxochromen-4-yl 4-(dimethylamino)-benzoate

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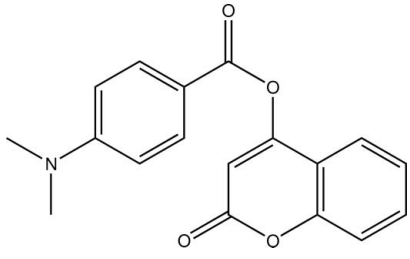
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.048; wR factor = 0.120; data-to-parameter ratio = 17.2.

In the title molecule, $C_{18}H_{15}NO_4$, the benzoate ring is oriented at a dihedral angle of 43.43 (6)° with respect to the planar [maximum deviation = 0.038 (2) Å] chromene ring. The crystal structure features $R_2^2(12)$ centrosymetric dimers formed *via* C—H···O interactions and these dimeric aggregates are connected by C—H···π interactions.

Related literature

For the biological activity of coumarin derivatives, see: Ukhov *et al.* (2001); Abd Elhafez *et al.* (2003); Basanagouda *et al.* (2009); Liu *et al.* (2008); Trapkov *et al.* (1996); Vukovic *et al.* (2010); Emmanuel-Giota *et al.* (2001); Hamdi & Dixneuf (2007); Wang *et al.* (2001); Marchenko *et al.* (2006). For hydrogen-bond graph-set motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{18}H_{15}NO_4$
M_r = 309.32
Triclinic, $P\overline{1}$
a = 7.4939 (2) Å
b = 10.2361 (3) Å
c = 10.6620 (3) Å
α = 92.307 (3)°
β = 103.935 (1)°
γ = 109.852 (2)°
V = 739.92 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 K
0.50 × 0.40 × 0.30 mm

Data collection

Nonius KappaCCD diffractometer
8424 measured reflections
3590 independent reflections
2897 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.120
S = 0.98
3585 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*3 is the centroid of the C15–C18/C22/C23 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C9—H91···O8^{i}	0.96	2.49	3.449 (2)	171
C7—H71···*Cg*3^{ii}	0.95	2.84	3.429 (2)	121
C20—H202···*Cg*3^{iii}	0.99	2.91	3.777 (2)	146

Symmetry codes: (i) $-x, -y, -z-1$; (ii) $-x, -y+1, -z$; (iii) $-x+1, -y+1, -z+1$.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2767).

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supporting information

Acta Cryst. (2011). E**67**, o2269–o2270 [doi:10.1107/S1600536811030844]

2-Oxochromen-4-yl 4-(dimethylamino)benzoate

Akoun Abou, Abdoulaye Djandé, Bintou Sessouma, Adama Saba and Rita Kakou-Yao

S1. Comment

Coumarin constitutes one of the major classes of naturally occurring compounds, and interest in its chemistry continues unabated because of its usefulness as biologically active agents. It also represents the core structure of several molecules of pharmaceutical importance. Coumarin and its derivatives have been reported to serve as anti-bacterial (Ukhov *et al.*, 2001; Abd Elhafez *et al.*, 2003; Basanagouda *et al.*, 2009; Liu *et al.*, 2008), anti-oxidant (Trapkov *et al.*, 1996; Vukovic *et al.*, 2010), anti-inflammatory (Emmanuel-Giota *et al.*, 2001; Hamdi & Dixneuf, 2007), anti-coagulant (Hamdi *et al.*, 2007) and anti-tumour (Wang *et al.*, 2001; Marchenko *et al.*, 2006) agents. Therefore, the synthesis of new coumarin derivatives is of considerable interest. In order to study the influence of new substituents on the activity of the coumarin derivative, the title compound, (I), has been synthesized and in this paper, we present its molecular structure, Fig. 1.

In (I), the planar chromene ring system resulting from the two coupled rings (benzene and 3,6-dihydro-2*H*-pyran) is oriented with respect to the benzoate-benzene ring at a dihedral angle of 43.43 (6)°. Atoms O14, N19, C13 and C21 are 0.046 (1), 0.052 (1), 0.079 (2) and 0.077 (3) Å out of the plane of the benzoate-benzene ring, respectively, so, they are coplanar with this ring.

In the crystal structure, intermolecular C—H···O interactions (Table 1) link the molecules into centrosymmetric dimers through $R_2^2(12)$ ring motifs (Bernstein *et al.*, 1995) (Fig. 2). Two weak C—H···π interactions formed between the H71 and H202 atoms and the centroid *Cg*3 of the benzoate-benzene ring (Table 1 and Fig. 3) further stabilize the structure.

S2. Experimental

To a solution of 4.10^{-2} mole of paradimethylamino benzoyl chloride in 150 ml of dried tetrahydrofuran, was added 0.12 mole of dried triethylamine and 4.10^{-2} mole of 4-hydroxycoumarin by small portions over 30 min. The mixture was then refluxed for 3 h and poured in 300 ml of chloroform or dichloromethane. The solution was acidified with dilute hydrochloric acid until the pH was 2 or 3. The organic layer was extracted, washed with water, dried over $MgSO_4$ and the solvent removed. The crude product was recrystallized in chloroform. Colourless crystals of the title compound are obtained in a good yield: 82.6%; M.pt. 445 K.

S3. Refinement

The H-atoms were placed at calculated positions and were included in the refinement in the riding model approximation with C—H in the range of 0.94–0.99 Å, and with U_{iso}(H) = 1.2–1.5U_{eq}(C).

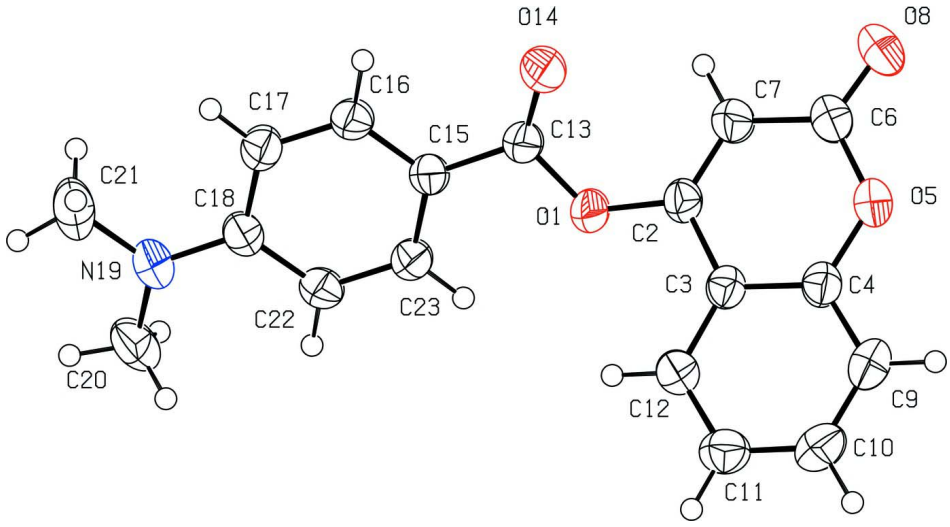

Figure 1

The molecular structure of (I) showing the atomic labeling scheme, with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

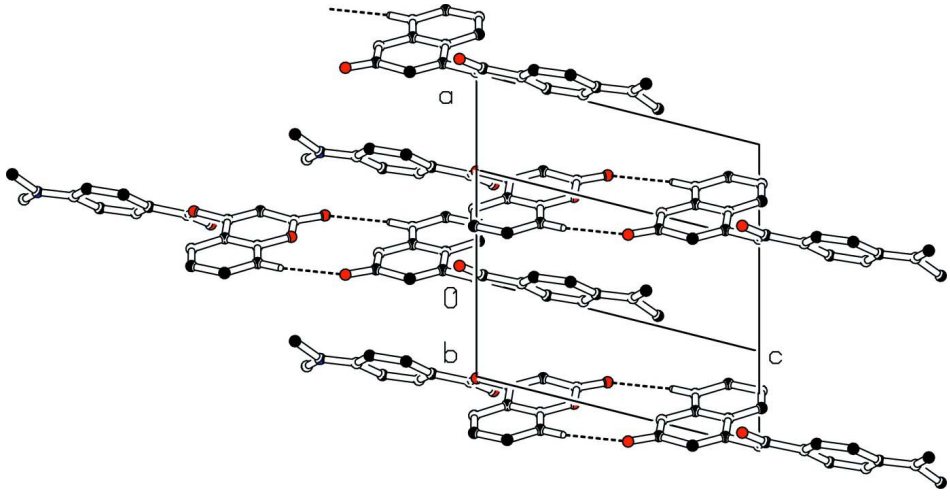

Figure 2

Crystal packing, viewed in projection down the *b* axis, showing parallel centrosymmetric dimers. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonds have been omitted for clarity.

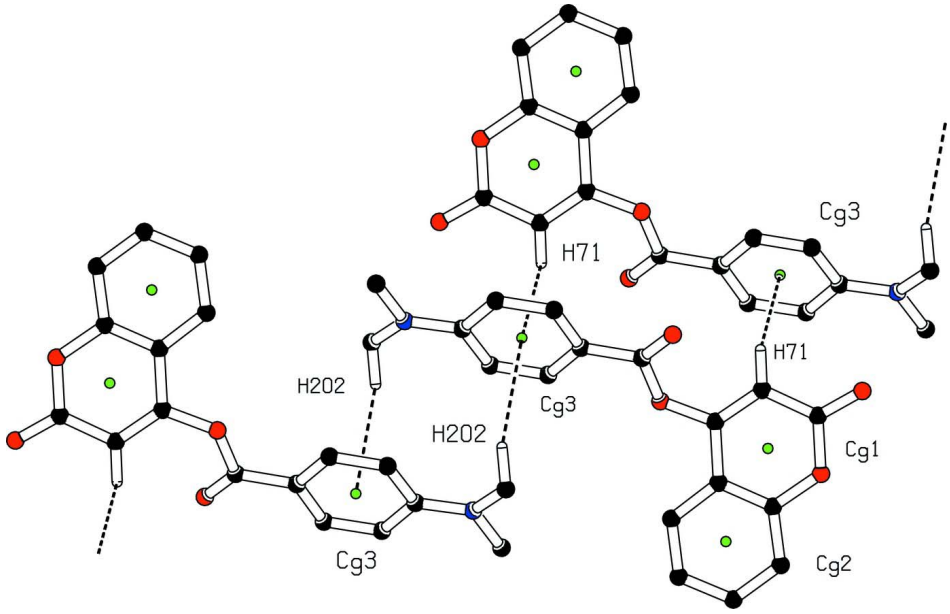

Figure 3

Crystal packing, showing C—H···π stacking interactions. The green dots are centroids of rings. H atoms not involved in C—H···π interactions have been omitted for clarity.

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Crystal data

$C_{18}H_{15}NO_4$
M_r = 309.32
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.4939 (2) Å
b = 10.2361 (3) Å
c = 10.6620 (3) Å
α = 92.307 (3)°
β = 103.935 (1)°
γ = 109.852 (2)°
V = 739.92 (4) Å^3

Z = 2
$F(000)$ = 324
D_x = 1.388 Mg m^{-3}
Melting point: 445 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8424 reflections
θ = 2.0–28.7°
μ = 0.10 mm^{-1}
T = 298 K
Parallelepiped, colourless
0.50 × 0.40 × 0.30 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
8424 measured reflections
3590 independent reflections

2897 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.7°, θ_{min} = 2.0°
h = −9→9
k = −13→13
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.120

S = 0.98
3585 reflections
208 parameters
0 restraints

60 constraints
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
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.05P)^2 + 0.22P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.00023$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

Refinement. The 5 reflections 1 0 0; 0 1 0; -1 0 1; 0 0 1; -1 1 1 have been measured with too low intensities. It might be caused by some systematical error, probably by shielding by a beam stop of these diffractions. They were not used in the refinement.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.16941 (15)	0.34698 (9)	−0.00111 (8)	0.0462
C2	0.13553 (19)	0.27238 (13)	−0.11979 (11)	0.0385
C3	0.16678 (17)	0.14123 (12)	−0.10953 (12)	0.0363
C4	0.12326 (18)	0.05485 (13)	−0.22545 (12)	0.0396
O5	0.05704 (15)	0.09384 (10)	−0.34457 (9)	0.0493
C6	0.0349 (2)	0.22168 (15)	−0.35487 (13)	0.0480
C7	0.0715 (2)	0.31027 (14)	−0.23575 (13)	0.0453
O8	−0.0149 (2)	0.24941 (13)	−0.46358 (10)	0.0703
C9	0.1466 (2)	−0.07411 (14)	−0.22593 (15)	0.0499
C10	0.2154 (2)	−0.11591 (15)	−0.10867 (16)	0.0531
C11	0.2611 (2)	−0.03116 (15)	0.00810 (15)	0.0504
C12	0.2373 (2)	0.09635 (14)	0.00783 (13)	0.0436
C13	0.26101 (19)	0.49181 (13)	0.02252 (12)	0.0394
O14	0.31022 (19)	0.56038 (11)	−0.06000 (10)	0.0605
C15	0.29273 (18)	0.54219 (12)	0.15893 (11)	0.0361
C16	0.40333 (19)	0.68335 (13)	0.20353 (12)	0.0399
C17	0.4486 (2)	0.73643 (14)	0.33261 (13)	0.0438
C18	0.38190 (19)	0.64988 (14)	0.42412 (12)	0.0399
N19	0.42952 (19)	0.70056 (13)	0.55279 (11)	0.0513
C20	0.3356 (3)	0.61733 (19)	0.64173 (14)	0.0591
C21	0.5486 (4)	0.8446 (2)	0.59983 (17)	0.0903
C22	0.2647 (2)	0.50796 (14)	0.37750 (12)	0.0422
C23	0.22417 (19)	0.45612 (13)	0.24879 (12)	0.0402
H71	0.0500	0.3963	−0.2441	0.0557*
H91	0.1132	−0.1320	−0.3079	0.0600*
H101	0.2329	−0.2053	−0.1062	0.0641*
H111	0.3097	−0.0605	0.0902	0.0601*
H121	0.2673	0.1552	0.0875	0.0528*
H161	0.4492	0.7442	0.1416	0.0485*
H171	0.5265	0.8322	0.3594	0.0528*
H201	0.3856	0.6702	0.7280	0.0885*
H203	0.1930	0.5921	0.6121	0.0885*
H202	0.3622	0.5286	0.6450	0.0885*

H211	0.5837	0.8563	0.6932	0.1350*
H213	0.4752	0.9046	0.5672	0.1350*
H212	0.6655	0.8711	0.5708	0.1350*
H221	0.2121	0.4466	0.4367	0.0519*
H231	0.1484	0.3589	0.2193	0.0481*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0692 (6)	0.0362 (5)	0.0336 (4)	0.0168 (4)	0.0184 (4)	0.0015 (4)
C2	0.0442 (7)	0.0376 (6)	0.0332 (6)	0.0136 (5)	0.0122 (5)	0.0005 (5)
C3	0.0355 (6)	0.0348 (6)	0.0363 (6)	0.0096 (5)	0.0109 (5)	0.0019 (5)
C4	0.0397 (6)	0.0371 (6)	0.0375 (6)	0.0095 (5)	0.0095 (5)	0.0001 (5)
O5	0.0631 (6)	0.0452 (5)	0.0345 (5)	0.0183 (5)	0.0072 (4)	−0.0028 (4)
C6	0.0555 (8)	0.0498 (8)	0.0358 (6)	0.0196 (6)	0.0069 (6)	0.0016 (6)
C7	0.0571 (8)	0.0444 (7)	0.0378 (6)	0.0241 (6)	0.0105 (6)	0.0042 (5)
O8	0.1030 (10)	0.0720 (8)	0.0340 (5)	0.0396 (7)	0.0038 (5)	0.0047 (5)
C9	0.0537 (8)	0.0389 (7)	0.0534 (8)	0.0137 (6)	0.0140 (6)	−0.0039 (6)
C10	0.0543 (8)	0.0396 (7)	0.0668 (9)	0.0197 (6)	0.0150 (7)	0.0058 (6)
C11	0.0500 (8)	0.0478 (8)	0.0534 (8)	0.0194 (6)	0.0104 (6)	0.0122 (6)
C12	0.0467 (7)	0.0439 (7)	0.0382 (6)	0.0149 (6)	0.0099 (5)	0.0052 (5)
C13	0.0487 (7)	0.0361 (6)	0.0357 (6)	0.0171 (5)	0.0133 (5)	0.0045 (5)
O14	0.0936 (8)	0.0462 (6)	0.0392 (5)	0.0159 (5)	0.0266 (5)	0.0087 (4)
C15	0.0419 (6)	0.0361 (6)	0.0331 (6)	0.0161 (5)	0.0124 (5)	0.0044 (5)
C16	0.0461 (7)	0.0368 (6)	0.0379 (6)	0.0129 (5)	0.0159 (5)	0.0076 (5)
C17	0.0477 (7)	0.0367 (6)	0.0415 (7)	0.0090 (5)	0.0120 (5)	0.0012 (5)
C18	0.0423 (7)	0.0456 (7)	0.0324 (6)	0.0185 (5)	0.0077 (5)	0.0033 (5)
N19	0.0632 (8)	0.0545 (7)	0.0319 (5)	0.0193 (6)	0.0086 (5)	0.0011 (5)
C20	0.0716 (10)	0.0777 (11)	0.0347 (7)	0.0325 (9)	0.0179 (7)	0.0104 (7)
C21	0.1312 (19)	0.0643 (11)	0.0409 (9)	0.0037 (11)	0.0096 (10)	−0.0089 (8)
C22	0.0506 (7)	0.0418 (7)	0.0366 (6)	0.0160 (6)	0.0163 (5)	0.0102 (5)
C23	0.0474 (7)	0.0344 (6)	0.0385 (6)	0.0128 (5)	0.0139 (5)	0.0048 (5)

Geometric parameters (Å, º)

O1—C2	1.3728 (14)	C13—C15	1.4586 (16)
O1—C13	1.3885 (15)	C15—C16	1.3937 (17)
C2—C3	1.4430 (17)	C15—C23	1.3985 (17)
C2—C7	1.3373 (18)	C16—C17	1.3762 (17)
C3—C4	1.3920 (16)	C16—H161	0.969
C3—C12	1.3958 (17)	C17—C18	1.4110 (18)
C4—O5	1.3749 (15)	C17—H171	0.944
C4—C9	1.3892 (18)	C18—N19	1.3637 (16)
O5—C6	1.3794 (17)	C18—C22	1.4121 (18)
C6—C7	1.4425 (18)	N19—C20	1.4490 (19)
C6—O8	1.2056 (16)	N19—C21	1.433 (2)
C7—H71	0.952	C20—H201	0.967
C9—C10	1.376 (2)	C20—H203	0.976

C9—H91	0.965	C20—H202	0.994
C10—C11	1.388 (2)	C21—H211	0.958
C10—H101	0.967	C21—H213	0.977
C11—C12	1.3764 (19)	C21—H212	0.956
C11—H111	0.965	C22—C23	1.3742 (17)
C12—H121	0.954	C22—H221	0.965
C13—O14	1.1970 (15)	C23—H231	0.958

C2—O1—C13	122.05 (10)	C13—C15—C23	123.81 (11)
O1—C2—C3	113.36 (10)	C16—C15—C23	117.98 (11)
O1—C2—C7	125.23 (12)	C15—C16—C17	121.34 (11)
C3—C2—C7	121.35 (11)	C15—C16—H161	118.6
C2—C3—C4	117.02 (11)	C17—C16—H161	120.1
C2—C3—C12	124.46 (11)	C16—C17—C18	120.99 (12)
C4—C3—C12	118.52 (12)	C16—C17—H171	118.8
C3—C4—O5	121.54 (11)	C18—C17—H171	120.2
C3—C4—C9	121.44 (12)	C17—C18—N19	121.52 (12)
O5—C4—C9	117.02 (11)	C17—C18—C22	117.38 (11)
C4—O5—C6	121.54 (10)	N19—C18—C22	121.10 (12)
O5—C6—C7	117.73 (11)	C18—N19—C20	120.85 (12)
O5—C6—O8	116.75 (12)	C18—N19—C21	120.98 (13)
C7—C6—O8	125.52 (14)	C20—N19—C21	117.41 (13)
C6—C7—C2	120.70 (12)	N19—C20—H201	109.4
C6—C7—H71	116.9	N19—C20—H203	110.3
C2—C7—H71	122.4	H201—C20—H203	109.8
C4—C9—C10	118.74 (13)	N19—C20—H202	110.5
C4—C9—H91	119.3	H201—C20—H202	109.5
C10—C9—H91	121.9	H203—C20—H202	107.3
C9—C10—C11	120.87 (13)	N19—C21—H211	108.5
C9—C10—H101	120.4	N19—C21—H213	110.0
C11—C10—H101	118.7	H211—C21—H213	109.9
C10—C11—C12	120.06 (13)	N19—C21—H212	110.5
C10—C11—H111	120.8	H211—C21—H212	109.4
C12—C11—H111	119.2	H213—C21—H212	108.5
C3—C12—C11	120.37 (12)	C18—C22—C23	120.82 (12)
C3—C12—H121	118.8	C18—C22—H221	119.6
C11—C12—H121	120.8	C23—C22—H221	119.6
O1—C13—O14	122.43 (11)	C15—C23—C22	121.45 (12)
O1—C13—C15	110.44 (10)	C15—C23—H231	118.7
O14—C13—C15	127.08 (12)	C22—C23—H231	119.9
C13—C15—C16	118.18 (11)		

Hydrogen-bond geometry (Å, º)

Cg3 is the centroid of the benzoate-benzene ring (C15–C18/C22/C23).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H91···O8^{i}	0.96	2.49	3.449 (2)	171

C7—H71···*Cg*3^{ii}	0.95	2.84	3.429 (2)	121
C20—H202···*Cg*3^{iii}	0.99	2.91	3.777 (2)	146

Symmetry codes: (i) −*x*, −*y*, −*z*−1; (ii) −*x*, −*y*+1, −*z*; (iii) −*x*+1, −*y*+1, −*z*+1.