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2,6-Bis(4-bromobenzylidene)cyclohexanone

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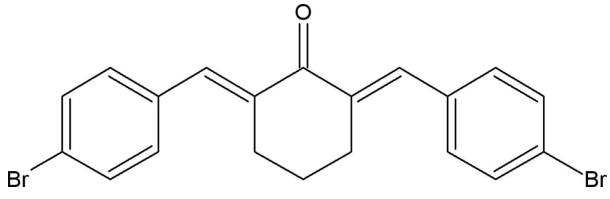
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.009 Å; R factor = 0.050; wR factor = 0.137; data-to-parameter ratio = 14.5.

The title compound, $C_{20}H_{16}Br_2O$, was prepared by the reaction of *p*-bromobenzophenone and cyclohexanone. There are three independent molecules in the asymmetric unit. Weak intermolecular C—H···O hydrogen bonds help stabilize the crystal structure.

Related literature

For synthetic background, see: Deli *et al.* (1984). The bond lengths in the title structure are comparable to those in some related structures (Yu *et al.*, 2000; Jia *et al.*, 1989; Butcher *et al.*, 2006).



Experimental

Crystal data

$C_{20}H_{16}Br_2O$
M_r = 432.15
Triclinic, $P\overline{1}$
a = 10.440 (2) Å
b = 15.724 (3) Å
c = 16.426 (4) Å
α = 75.703 (3)°
β = 87.057 (3)°
γ = 89.122 (4)°
V = 2609.4 (9) Å^3
Z = 6
Mo $K\alpha$ radiation
μ = 4.66 mm^{-1}
T = 298 (2) K
0.50 × 0.25 × 0.21 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.204, T_{max} = 0.441
(expected range = 0.174–0.376)
13234 measured reflections
9006 independent reflections
4543 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.137
S = 1.00
9006 reflections
622 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C27—H27···O2^{i}	0.93	2.58	3.310 (8)	136
C14—H14···O3^{ii}	0.93	2.59	3.446 (8)	154

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+2, -y+1, -z$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2405).

References

Bruker (1997). *SMART* (Version 5.044), *SAINT* (Version 5.01) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Butcher, R. J., Yathirajan, H. S., Sarojini, B. K., Narayana, B. & Indira, J. (2006). *Acta Cryst.* E**62**, o1910–o1912.
Deli, J., Lorand, T., Szabo, D. & Foldesi, A. (1984). *Pharmazie*, **39**, 539–544.
Jia, Z., Quail, J. W., Arora, V. K. & Dimmock, J. R. (1989). *Acta Cryst.* C**45**, 285–289.
Sheldrick, G. M. (1996). *SADABS*. Version 2.01. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Yu, R. C., Yakimansky, A. V., Kothe, H., Voigt-Martin, I. G., Schollmeyer, D., Jansen, J., Zandbergen, H. & Tenkovtsev, A. V. (2000). *Acta Cryst.* A**56**, 436–450.

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supplementary materials

Acta Cryst. (2007). E**63**, o3113 [**doi:10.1107/S1600536807026943**]

2,6-Bis(4-bromobenzylidene)cyclohexanone

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Comment

As useful precursors to potentially bioactive pyrimidine derivatives, a,a-bis(substituted benzylidene) cycloalkanones have attracted considerable attention for many years(Deli *et al.*, 1984). In recent years, a series of non-linear optically active bis(benzylidene) ketones have been synthesized (Yu *et al.*, 2002). As part of our search for new non-linear optically active compounds we synthesized the title compound (I), and describe its structure here.

The asymmetric unit of the title compound contains three molecules in the asymmetric unit (Fig. 1). In each molecule all of the bond lengthes and bond angles fall in the normal range(Yu *et al.*, 2002; Jia *et al.*, 1989; Butcher *et al.*, 2006). The crystal structure is stabilized by weak intermolecular C—H···O intramolecular hydrogen bonds.

Experimental

A mixture of the *p*-Bromobenzophenone (0.2 mol), and cyclohexanone (0.1 mol) and 10% NaOH(10 ml) was stirred in ethanol (30 mL) for 5 h to afford the title compound (0.08 mol, yield 80%). Single crystals suitable for X-ray measurements were obtained by recrystallization of the title compound from ethanol at room temperature.

Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances=0.93–0.97 Å, and with U_{iso}=1.2(C).

Figures

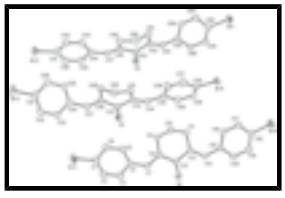


Fig. 1. The asymmetric unit of the title crystal structure showing 30% probability displacement ellipsoids and the atom-numbering scheme.

2,6-Bis(4-bromobenzylidene)cyclohexanone

Crystal data

$C_{20}H_{16}Br_2O$	Z = 6
M_r = 432.15	F_{000} = 1284
Triclinic, $P\overline{1}$	D_x = 1.650 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation

	λ = 0.71073 Å
a = 10.440 (2) Å	Cell parameters from 2666 reflections
b = 15.724 (3) Å	θ = 2.4–22.2º
c = 16.426 (4) Å	μ = 4.66 mm^{-1}
α = 75.703 (3)º	*T* = 298 (2) K
β = 87.057 (3)º	Bar, yellow
γ = 89.122 (4)º	0.50 × 0.25 × 0.21 mm
V = 2609.4 (9) $Å^3$	

Data collection

Bruker SMART CCD diffractometer	9006 independent reflections
Radiation source: fine-focus sealed tube	4543 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.042
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.3º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −12→11
T_{min} = 0.204, T_{max} = 0.441	*k* = −18→15
13234 measured reflections	*l* = −19→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.050	H-atom parameters constrained
$wR(F^2)$ = 0.137	$w = 1/[\sigma^2(F_o^2) + (0.0411P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max}$ = 0.001
9006 reflections	$\Delta\rho_{max}$ = 0.45 e $Å^{-3}$
622 parameters	$\Delta\rho_{min}$ = −0.64 e $Å^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.1088 (6)	0.4347 (5)	0.3849 (4)	0.0523 (17)
C2	1.1209 (6)	0.3535 (4)	0.3690 (4)	0.0587 (19)
H2	1.1587	0.3077	0.4074	0.070*
C3	1.0761 (6)	0.3401 (4)	0.2949 (4)	0.0544 (18)
H3	1.0861	0.2854	0.2834	0.065*
C4	1.0163 (5)	0.4079 (4)	0.2375 (4)	0.0413 (15)
C5	1.0041 (6)	0.4886 (4)	0.2566 (4)	0.0501 (17)
H5	0.9637	0.5344	0.2196	0.060*
C6	1.0506 (6)	0.5029 (4)	0.3294 (4)	0.0548 (18)
H6	1.0426	0.5579	0.3409	0.066*
C7	0.9749 (6)	0.3909 (4)	0.1589 (4)	0.0461 (16)
H7	1.0195	0.3465	0.1413	0.055*
C8	0.8836 (6)	0.4293 (3)	0.1092 (4)	0.0428 (16)
C9	0.7937 (6)	0.4981 (4)	0.1271 (4)	0.0544 (18)
H9A	0.8329	0.5554	0.1061	0.065*
H9B	0.7785	0.4884	0.1874	0.065*
C10	0.6665 (6)	0.4970 (4)	0.0865 (4)	0.0546 (18)
H10A	0.6259	0.4403	0.1080	0.065*
H10B	0.6099	0.5415	0.1002	0.065*
C11	0.6885 (6)	0.5145 (4)	−0.0071 (4)	0.0511 (17)
H11A	0.7263	0.5722	−0.0284	0.061*
H11B	0.6068	0.5145	−0.0326	0.061*
C12	0.7763 (5)	0.4462 (3)	−0.0319 (4)	0.0400 (15)
C13	0.8705 (6)	0.3999 (4)	0.0307 (4)	0.0461 (16)
C14	0.7722 (6)	0.4218 (4)	−0.1034 (4)	0.0439 (16)
H14	0.8283	0.3768	−0.1086	0.053*
C15	0.6925 (5)	0.4552 (4)	−0.1758 (4)	0.0400 (15)
C16	0.6434 (6)	0.5399 (4)	−0.1979 (4)	0.0475 (16)
H16	0.6622	0.5796	−0.1666	0.057*
C17	0.5679 (6)	0.5655 (4)	−0.2650 (4)	0.0549 (18)
H17	0.5364	0.6226	−0.2794	0.066*
C18	0.5379 (6)	0.5078 (4)	−0.3117 (4)	0.0499 (17)
C19	0.5880 (6)	0.4244 (4)	−0.2933 (4)	0.0549 (18)
H19	0.5701	0.3858	−0.3259	0.066*
C20	0.6650 (6)	0.3987 (4)	−0.2260 (4)	0.0533 (18)
H20	0.6994	0.3424	−0.2137	0.064*
C21	1.1353 (6)	0.8084 (5)	0.5762 (4)	0.0582 (19)
C22	1.1976 (7)	0.7428 (5)	0.5499 (5)	0.068 (2)
H22	1.2605	0.7101	0.5820	0.081*
C23	1.1671 (6)	0.7252 (4)	0.4759 (4)	0.061 (2)
H23	1.2105	0.6803	0.4583	0.073*
C24	1.0732 (6)	0.7722 (4)	0.4260 (4)	0.0480 (17)
C25	1.0136 (6)	0.8391 (4)	0.4543 (4)	0.0532 (18)
H25	0.9511	0.8722	0.4222	0.064*
C26	1.0432 (7)	0.8588 (4)	0.5287 (4)	0.0604 (19)

H26	1.0023	0.9046	0.5463	0.073*
C27	1.0465 (6)	0.7510 (4)	0.3464 (4)	0.0473 (16)
H27	1.1175	0.7357	0.3164	0.057*
C28	0.9338 (6)	0.7506 (3)	0.3113 (4)	0.0401 (15)
C29	0.8060 (6)	0.7644 (4)	0.3515 (4)	0.0479 (16)
H29A	0.7790	0.8249	0.3307	0.058*
H29B	0.8126	0.7532	0.4119	0.058*
C30	0.7079 (6)	0.7033 (4)	0.3317 (4)	0.0543 (18)
H30A	0.6267	0.7097	0.3612	0.065*
H30B	0.7364	0.6430	0.3511	0.065*
C31	0.6895 (6)	0.7242 (4)	0.2374 (4)	0.0518 (17)
H31A	0.6370	0.6787	0.2256	0.062*
H31B	0.6430	0.7792	0.2211	0.062*
C32	0.8106 (5)	0.7311 (3)	0.1851 (4)	0.0369 (15)
C33	0.9362 (6)	0.7302 (3)	0.2272 (4)	0.0391 (15)
C34	0.8204 (6)	0.7397 (3)	0.1024 (4)	0.0432 (16)
H34	0.9042	0.7412	0.0801	0.052*
C35	0.7229 (6)	0.7471 (3)	0.0405 (4)	0.0431 (16)
C36	0.5898 (7)	0.7484 (5)	0.0563 (5)	0.074 (2)
H36	0.5571	0.7460	0.1106	0.089*
C37	0.5059 (7)	0.7533 (5)	−0.0075 (5)	0.076 (2)
H37	0.4178	0.7536	0.0043	0.091*
C38	0.5527 (7)	0.7578 (4)	−0.0872 (4)	0.0483 (17)
C39	0.6811 (7)	0.7597 (4)	−0.1056 (4)	0.0553 (18)
H39	0.7125	0.7645	−0.1606	0.066*
C40	0.7642 (6)	0.7545 (4)	−0.0425 (4)	0.0501 (17)
H40	0.8519	0.7561	−0.0559	0.060*
C41	0.6957 (6)	0.9087 (5)	0.5897 (4)	0.0558 (18)
C42	0.6926 (6)	0.9723 (4)	0.5169 (5)	0.0596 (19)
H42	0.7352	1.0250	0.5122	0.071*
C43	0.6273 (6)	0.9600 (4)	0.4499 (5)	0.062 (2)
H43	0.6266	1.0046	0.4007	0.075*
C44	0.5628 (6)	0.8826 (4)	0.4544 (4)	0.0503 (17)
C45	0.5667 (7)	0.8193 (4)	0.5308 (5)	0.068 (2)
H45	0.5240	0.7665	0.5365	0.081*
C46	0.6315 (7)	0.8321 (5)	0.5981 (5)	0.068 (2)
H46	0.6313	0.7889	0.6484	0.082*
C47	0.4893 (6)	0.8628 (4)	0.3875 (4)	0.0487 (17)
H47	0.4360	0.8142	0.4058	0.058*
C48	0.4843 (6)	0.9008 (4)	0.3060 (4)	0.0445 (16)
C49	0.5611 (6)	0.9797 (4)	0.2571 (4)	0.0543 (18)
H49A	0.5256	1.0320	0.2710	0.065*
H49B	0.6488	0.9733	0.2748	0.065*
C50	0.5624 (6)	0.9919 (4)	0.1636 (4)	0.0580 (18)
H50A	0.6091	0.9439	0.1483	0.070*
H50B	0.6062	1.0461	0.1360	0.070*
C51	0.4273 (6)	0.9948 (4)	0.1339 (4)	0.0528 (17)
H51A	0.3810	1.0433	0.1486	0.063*
H51B	0.4304	1.0048	0.0731	0.063*

C52	0.3572 (6)	0.9102 (4)	0.1731 (4)	0.0436 (16)
C53	0.3959 (6)	0.8600 (4)	0.2575 (4)	0.0434 (16)
C54	0.2660 (6)	0.8759 (4)	0.1362 (4)	0.0461 (16)
H54	0.2337	0.8221	0.1669	0.055*
C55	0.2108 (5)	0.9103 (4)	0.0558 (4)	0.0403 (15)
C56	0.1940 (6)	0.9985 (4)	0.0184 (4)	0.0504 (17)
H56	0.2198	1.0396	0.0464	0.060*
C57	0.1411 (6)	1.0280 (4)	−0.0579 (4)	0.0540 (18)
H57	0.1296	1.0877	−0.0809	0.065*
C58	0.1052 (6)	0.9670 (4)	−0.1001 (4)	0.0490 (17)
C59	0.1171 (6)	0.8782 (4)	−0.0650 (4)	0.0513 (17)
H59	0.0916	0.8376	−0.0936	0.062*
C60	0.1675 (5)	0.8508 (4)	0.0130 (4)	0.0475 (17)
H60	0.1729	0.7910	0.0378	0.057*
Br1	0.03242 (8)	1.00691 (5)	−0.20624 (5)	0.0754 (3)
Br2	0.78651 (7)	0.92869 (5)	0.67997 (5)	0.0762 (3)
Br3	1.17664 (9)	0.45461 (5)	0.48307 (5)	0.0865 (3)
Br4	0.43065 (9)	0.54396 (5)	−0.40375 (6)	0.0902 (3)
Br5	1.17811 (8)	0.83500 (6)	0.67812 (5)	0.0891 (3)
Br6	0.43509 (8)	0.76421 (5)	−0.17299 (5)	0.0735 (3)
O1	0.9370 (5)	0.3406 (3)	0.0155 (3)	0.0764 (16)
O2	0.3535 (4)	0.7851 (3)	0.2883 (3)	0.0568 (12)
O3	1.0369 (4)	0.7152 (3)	0.1924 (3)	0.0545 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.045 (4)	0.069 (5)	0.038 (4)	−0.010 (3)	−0.001 (3)	−0.004 (4)
C2	0.063 (5)	0.059 (5)	0.047 (5)	0.003 (4)	−0.011 (4)	0.002 (4)
C3	0.061 (5)	0.044 (4)	0.054 (5)	0.001 (3)	−0.004 (4)	−0.004 (3)
C4	0.036 (4)	0.044 (4)	0.037 (4)	−0.002 (3)	0.004 (3)	0.001 (3)
C5	0.066 (5)	0.045 (4)	0.039 (4)	0.006 (3)	−0.012 (4)	−0.008 (3)
C6	0.066 (5)	0.047 (4)	0.050 (5)	0.002 (3)	−0.008 (4)	−0.006 (3)
C7	0.052 (4)	0.043 (4)	0.045 (4)	0.005 (3)	0.001 (4)	−0.014 (3)
C8	0.042 (4)	0.037 (3)	0.049 (4)	0.001 (3)	−0.003 (3)	−0.009 (3)
C9	0.061 (5)	0.059 (4)	0.047 (4)	0.018 (3)	−0.007 (4)	−0.020 (3)
C10	0.049 (4)	0.065 (4)	0.051 (5)	0.018 (3)	0.002 (4)	−0.020 (3)
C11	0.053 (4)	0.049 (4)	0.052 (5)	0.012 (3)	−0.005 (4)	−0.012 (3)
C12	0.040 (4)	0.035 (3)	0.045 (4)	0.000 (3)	0.003 (3)	−0.012 (3)
C13	0.045 (4)	0.049 (4)	0.047 (4)	0.004 (3)	0.002 (3)	−0.019 (3)
C14	0.044 (4)	0.040 (3)	0.048 (4)	0.003 (3)	0.001 (3)	−0.012 (3)
C15	0.042 (4)	0.040 (4)	0.037 (4)	−0.002 (3)	0.002 (3)	−0.009 (3)
C16	0.059 (4)	0.045 (4)	0.039 (4)	−0.005 (3)	0.002 (4)	−0.014 (3)
C17	0.065 (5)	0.045 (4)	0.052 (5)	0.007 (3)	−0.001 (4)	−0.007 (3)
C18	0.046 (4)	0.056 (4)	0.047 (4)	0.000 (3)	−0.012 (3)	−0.009 (3)
C19	0.061 (5)	0.052 (4)	0.056 (5)	−0.007 (4)	−0.003 (4)	−0.022 (3)
C20	0.054 (4)	0.046 (4)	0.063 (5)	0.007 (3)	−0.005 (4)	−0.019 (3)
C21	0.046 (4)	0.080 (5)	0.053 (5)	−0.009 (4)	−0.004 (4)	−0.025 (4)

C22	0.049 (5)	0.088 (5)	0.070 (6)	0.010 (4)	−0.017 (4)	−0.025 (4)
C23	0.050 (4)	0.076 (5)	0.063 (5)	0.018 (4)	−0.015 (4)	−0.027 (4)
C24	0.045 (4)	0.046 (4)	0.052 (5)	−0.007 (3)	0.003 (4)	−0.011 (3)
C25	0.054 (4)	0.054 (4)	0.055 (5)	0.004 (3)	−0.010 (4)	−0.019 (4)
C26	0.062 (5)	0.059 (4)	0.066 (5)	−0.005 (4)	0.006 (4)	−0.028 (4)
C27	0.043 (4)	0.054 (4)	0.046 (4)	−0.001 (3)	0.006 (3)	−0.016 (3)
C28	0.041 (4)	0.039 (3)	0.038 (4)	0.003 (3)	0.005 (3)	−0.007 (3)
C29	0.045 (4)	0.058 (4)	0.040 (4)	0.000 (3)	0.010 (3)	−0.015 (3)
C30	0.047 (4)	0.066 (4)	0.045 (5)	−0.011 (3)	0.014 (4)	−0.008 (3)
C31	0.046 (4)	0.063 (4)	0.045 (5)	0.003 (3)	−0.003 (4)	−0.011 (3)
C32	0.035 (4)	0.032 (3)	0.041 (4)	0.005 (3)	−0.001 (3)	−0.005 (3)
C33	0.048 (4)	0.033 (3)	0.035 (4)	0.001 (3)	0.002 (3)	−0.007 (3)
C34	0.051 (4)	0.040 (3)	0.039 (4)	0.003 (3)	0.002 (3)	−0.011 (3)
C35	0.049 (4)	0.035 (3)	0.043 (4)	0.004 (3)	−0.003 (4)	−0.007 (3)
C36	0.059 (5)	0.121 (6)	0.042 (5)	0.014 (4)	−0.001 (4)	−0.019 (4)
C37	0.046 (5)	0.120 (6)	0.062 (6)	0.015 (4)	−0.004 (4)	−0.024 (5)
C38	0.063 (5)	0.039 (4)	0.044 (5)	0.012 (3)	−0.018 (4)	−0.009 (3)
C39	0.070 (5)	0.058 (4)	0.042 (4)	0.006 (4)	−0.004 (4)	−0.020 (3)
C40	0.047 (4)	0.053 (4)	0.051 (5)	−0.002 (3)	0.004 (4)	−0.016 (3)
C41	0.046 (4)	0.069 (5)	0.051 (5)	0.000 (4)	−0.001 (4)	−0.014 (4)
C42	0.057 (5)	0.055 (4)	0.068 (6)	−0.010 (3)	−0.007 (4)	−0.017 (4)
C43	0.064 (5)	0.048 (4)	0.071 (5)	−0.010 (4)	−0.016 (4)	−0.003 (4)
C44	0.041 (4)	0.048 (4)	0.061 (5)	0.000 (3)	0.004 (4)	−0.011 (4)
C45	0.076 (6)	0.059 (5)	0.065 (6)	−0.017 (4)	0.005 (5)	−0.011 (4)
C46	0.078 (6)	0.068 (5)	0.053 (5)	−0.007 (4)	0.004 (4)	−0.006 (4)
C47	0.039 (4)	0.043 (4)	0.067 (5)	−0.001 (3)	−0.003 (4)	−0.018 (4)
C48	0.034 (4)	0.041 (4)	0.057 (5)	0.009 (3)	−0.004 (3)	−0.009 (3)
C49	0.040 (4)	0.045 (4)	0.074 (6)	−0.005 (3)	−0.003 (4)	−0.006 (3)
C50	0.052 (5)	0.057 (4)	0.059 (5)	−0.010 (3)	0.013 (4)	−0.006 (3)
C51	0.052 (4)	0.056 (4)	0.046 (4)	−0.007 (3)	0.005 (4)	−0.006 (3)
C52	0.041 (4)	0.041 (4)	0.049 (4)	0.001 (3)	0.006 (3)	−0.011 (3)
C53	0.038 (4)	0.043 (4)	0.051 (4)	0.005 (3)	0.005 (3)	−0.017 (3)
C54	0.046 (4)	0.038 (3)	0.051 (4)	−0.003 (3)	0.002 (3)	−0.005 (3)
C55	0.032 (4)	0.046 (4)	0.039 (4)	0.001 (3)	0.007 (3)	−0.004 (3)
C56	0.054 (4)	0.042 (4)	0.055 (5)	0.002 (3)	0.003 (4)	−0.013 (3)
C57	0.064 (5)	0.040 (4)	0.053 (5)	0.002 (3)	0.004 (4)	−0.003 (3)
C58	0.039 (4)	0.056 (4)	0.049 (4)	0.004 (3)	0.012 (3)	−0.009 (3)
C59	0.045 (4)	0.051 (4)	0.062 (5)	−0.006 (3)	0.001 (4)	−0.022 (4)
C60	0.037 (4)	0.042 (4)	0.065 (5)	0.003 (3)	−0.002 (4)	−0.018 (3)
Br1	0.0843 (6)	0.0852 (5)	0.0542 (5)	0.0082 (4)	−0.0072 (4)	−0.0121 (4)
Br2	0.0684 (5)	0.1029 (6)	0.0606 (6)	0.0009 (4)	−0.0100 (4)	−0.0249 (4)
Br3	0.1052 (7)	0.1030 (6)	0.0499 (5)	−0.0011 (5)	−0.0273 (5)	−0.0116 (4)
Br4	0.1003 (7)	0.0886 (6)	0.0856 (7)	0.0159 (5)	−0.0451 (6)	−0.0221 (5)
Br5	0.0922 (7)	0.1209 (7)	0.0653 (6)	−0.0224 (5)	−0.0129 (5)	−0.0405 (5)
Br6	0.0905 (6)	0.0670 (5)	0.0674 (6)	0.0121 (4)	−0.0346 (5)	−0.0191 (4)
O1	0.083 (4)	0.080 (3)	0.083 (4)	0.046 (3)	−0.028 (3)	−0.051 (3)
O2	0.058 (3)	0.046 (3)	0.062 (3)	−0.010 (2)	−0.006 (3)	−0.005 (2)
O3	0.042 (3)	0.075 (3)	0.051 (3)	0.009 (2)	0.001 (2)	−0.026 (2)

Geometric parameters (Å, °)

C1—C2	1.367 (8)	C30—H30B	0.9700
C1—C6	1.381 (8)	C31—C32	1.482 (8)
C1—Br3	1.890 (6)	C31—H31A	0.9700
C2—C3	1.389 (8)	C31—H31B	0.9700
C2—H2	0.9300	C32—C34	1.331 (7)
C3—C4	1.400 (7)	C32—C33	1.511 (7)
C3—H3	0.9300	C33—O3	1.219 (7)
C4—C5	1.383 (7)	C34—C35	1.459 (7)
C4—C7	1.468 (8)	C34—H34	0.9300
C5—C6	1.384 (8)	C35—C40	1.385 (8)
C5—H5	0.9300	C35—C36	1.402 (9)
C6—H6	0.9300	C36—C37	1.387 (8)
C7—C8	1.326 (7)	C36—H36	0.9300
C7—H7	0.9300	C37—C38	1.360 (9)
C8—C13	1.486 (8)	C37—H37	0.9300
C8—C9	1.495 (8)	C38—C39	1.359 (9)
C9—C10	1.518 (7)	C38—Br6	1.899 (6)
C9—H9A	0.9700	C39—C40	1.373 (7)
C9—H9B	0.9700	C39—H39	0.9300
C10—C11	1.499 (8)	C40—H40	0.9300
C10—H10A	0.9700	C41—C42	1.357 (9)
C10—H10B	0.9700	C41—C46	1.362 (9)
C11—C12	1.520 (8)	C41—Br2	1.891 (6)
C11—H11A	0.9700	C42—C43	1.378 (8)
C11—H11B	0.9700	C42—H42	0.9300
C12—C14	1.325 (8)	C43—C44	1.385 (8)
C12—C13	1.505 (8)	C43—H43	0.9300
C13—O1	1.221 (6)	C44—C45	1.397 (9)
C14—C15	1.470 (7)	C44—C47	1.467 (8)
C14—H14	0.9300	C45—C46	1.383 (9)
C15—C16	1.389 (7)	C45—H45	0.9300
C15—C20	1.394 (8)	C46—H46	0.9300
C16—C17	1.364 (8)	C47—C48	1.328 (8)
C16—H16	0.9300	C47—H47	0.9300
C17—C18	1.374 (8)	C48—C53	1.499 (8)
C17—H17	0.9300	C48—C49	1.517 (8)
C18—C19	1.372 (8)	C49—C50	1.500 (9)
C18—Br4	1.895 (6)	C49—H49A	0.9700
C19—C20	1.376 (8)	C49—H49B	0.9700
C19—H19	0.9300	C50—C51	1.512 (8)
C20—H20	0.9300	C50—H50A	0.9700
C21—C22	1.359 (9)	C50—H50B	0.9700
C21—C26	1.382 (8)	C51—C52	1.509 (8)
C21—Br5	1.899 (6)	C51—H51A	0.9700
C22—C23	1.365 (9)	C51—H51B	0.9700
C22—H22	0.9300	C52—C54	1.342 (7)

C23—C24	1.393 (8)	C52—C53	1.489 (8)
C23—H23	0.9300	C53—O2	1.239 (6)
C24—C25	1.380 (8)	C54—C55	1.444 (7)
C24—C27	1.467 (8)	C54—H54	0.9300
C25—C26	1.383 (8)	C55—C56	1.384 (7)
C25—H25	0.9300	C55—C60	1.394 (8)
C26—H26	0.9300	C56—C57	1.366 (8)
C27—C28	1.338 (7)	C56—H56	0.9300
C27—H27	0.9300	C57—C58	1.381 (8)
C28—C33	1.492 (8)	C57—H57	0.9300
C28—C29	1.494 (8)	C58—C59	1.378 (8)
C29—C30	1.516 (7)	C58—Br1	1.895 (6)
C29—H29A	0.9700	C59—C60	1.376 (8)
C29—H29B	0.9700	C59—H59	0.9300
C30—C31	1.524 (8)	C60—H60	0.9300
C30—H30A	0.9700		
C2—C1—C6	121.1 (6)	H30A—C30—H30B	108.1
C2—C1—Br3	119.5 (5)	C32—C31—C30	114.2 (5)
C6—C1—Br3	119.4 (5)	C32—C31—H31A	108.7
C1—C2—C3	119.4 (6)	C30—C31—H31A	108.7
C1—C2—H2	120.3	C32—C31—H31B	108.7
C3—C2—H2	120.3	C30—C31—H31B	108.7
C2—C3—C4	120.9 (6)	H31A—C31—H31B	107.6
C2—C3—H3	119.5	C34—C32—C31	125.8 (5)
C4—C3—H3	119.5	C34—C32—C33	115.5 (6)
C5—C4—C3	117.9 (6)	C31—C32—C33	118.7 (5)
C5—C4—C7	123.5 (5)	O3—C33—C28	121.0 (5)
C3—C4—C7	118.6 (5)	O3—C33—C32	120.8 (5)
C4—C5—C6	121.5 (5)	C28—C33—C32	118.2 (6)
C4—C5—H5	119.3	C32—C34—C35	131.5 (6)
C6—C5—H5	119.3	C32—C34—H34	114.3
C1—C6—C5	119.2 (6)	C35—C34—H34	114.3
C1—C6—H6	120.4	C40—C35—C36	116.0 (6)
C5—C6—H6	120.4	C40—C35—C34	117.7 (6)
C8—C7—C4	129.9 (6)	C36—C35—C34	126.3 (6)
C8—C7—H7	115.1	C37—C36—C35	121.2 (7)
C4—C7—H7	115.1	C37—C36—H36	119.4
C7—C8—C13	116.5 (5)	C35—C36—H36	119.4
C7—C8—C9	124.9 (6)	C38—C37—C36	119.9 (7)
C13—C8—C9	118.6 (5)	C38—C37—H37	120.1
C8—C9—C10	111.8 (5)	C36—C37—H37	120.1
C8—C9—H9A	109.3	C39—C38—C37	120.7 (6)
C10—C9—H9A	109.3	C39—C38—Br6	120.6 (5)
C8—C9—H9B	109.3	C37—C38—Br6	118.7 (6)
C10—C9—H9B	109.3	C38—C39—C40	119.5 (6)
H9A—C9—H9B	107.9	C38—C39—H39	120.3
C11—C10—C9	109.7 (6)	C40—C39—H39	120.3
C11—C10—H10A	109.7	C39—C40—C35	122.7 (7)
C9—C10—H10A	109.7	C39—C40—H40	118.6

C11—C10—H10B	109.7	C35—C40—H40	118.6
C9—C10—H10B	109.7	C42—C41—C46	120.1 (6)
H10A—C10—H10B	108.2	C42—C41—Br2	119.4 (5)
C10—C11—C12	111.8 (5)	C46—C41—Br2	120.4 (6)
C10—C11—H11A	109.3	C41—C42—C43	121.0 (6)
C12—C11—H11A	109.3	C41—C42—H42	119.5
C10—C11—H11B	109.3	C43—C42—H42	119.5
C12—C11—H11B	109.3	C42—C43—C44	121.3 (6)
H11A—C11—H11B	107.9	C42—C43—H43	119.3
C14—C12—C13	117.6 (5)	C44—C43—H43	119.3
C14—C12—C11	124.6 (5)	C43—C44—C45	116.0 (6)
C13—C12—C11	117.7 (5)	C43—C44—C47	125.9 (6)
O1—C13—C8	121.1 (5)	C45—C44—C47	118.1 (6)
O1—C13—C12	119.8 (5)	C46—C45—C44	122.6 (6)
C8—C13—C12	119.1 (5)	C46—C45—H45	118.7
C12—C14—C15	129.9 (6)	C44—C45—H45	118.7
C12—C14—H14	115.1	C41—C46—C45	119.0 (7)
C15—C14—H14	115.1	C41—C46—H46	120.5
C16—C15—C20	117.7 (5)	C45—C46—H46	120.5
C16—C15—C14	124.1 (5)	C48—C47—C44	132.4 (6)
C20—C15—C14	118.3 (5)	C48—C47—H47	113.8
C17—C16—C15	120.7 (5)	C44—C47—H47	113.8
C17—C16—H16	119.7	C47—C48—C53	116.4 (6)
C15—C16—H16	119.7	C47—C48—C49	126.6 (6)
C16—C17—C18	120.7 (6)	C53—C48—C49	117.0 (6)
C16—C17—H17	119.7	C50—C49—C48	113.6 (5)
C18—C17—H17	119.7	C50—C49—H49A	108.8
C19—C18—C17	120.2 (6)	C48—C49—H49A	108.8
C19—C18—Br4	119.7 (5)	C50—C49—H49B	108.8
C17—C18—Br4	120.1 (5)	C48—C49—H49B	108.8
C18—C19—C20	119.2 (6)	H49A—C49—H49B	107.7
C18—C19—H19	120.4	C49—C50—C51	110.8 (6)
C20—C19—H19	120.4	C49—C50—H50A	109.5
C19—C20—C15	121.6 (6)	C51—C50—H50A	109.5
C19—C20—H20	119.2	C49—C50—H50B	109.5
C15—C20—H20	119.2	C51—C50—H50B	109.5
C22—C21—C26	121.3 (6)	H50A—C50—H50B	108.1
C22—C21—Br5	120.0 (5)	C52—C51—C50	111.2 (5)
C26—C21—Br5	118.6 (6)	C52—C51—H51A	109.4
C21—C22—C23	119.4 (6)	C50—C51—H51A	109.4
C21—C22—H22	120.3	C52—C51—H51B	109.4
C23—C22—H22	120.3	C50—C51—H51B	109.4
C22—C23—C24	122.2 (6)	H51A—C51—H51B	108.0
C22—C23—H23	118.9	C54—C52—C53	118.0 (5)
C24—C23—H23	118.9	C54—C52—C51	124.8 (6)
C25—C24—C23	116.5 (6)	C53—C52—C51	117.2 (5)
C25—C24—C27	124.0 (6)	O2—C53—C52	120.2 (5)
C23—C24—C27	119.5 (6)	O2—C53—C48	119.9 (6)
C24—C25—C26	122.6 (6)	C52—C53—C48	119.9 (6)

C24—C25—H25	118.7	C52—C54—C55	129.4 (6)
C26—C25—H25	118.7	C52—C54—H54	115.3
C21—C26—C25	118.0 (6)	C55—C54—H54	115.3
C21—C26—H26	121.0	C56—C55—C60	116.9 (6)
C25—C26—H26	121.0	C56—C55—C54	124.9 (6)
C28—C27—C24	128.7 (6)	C60—C55—C54	118.1 (5)
C28—C27—H27	115.7	C57—C56—C55	122.8 (6)
C24—C27—H27	115.7	C57—C56—H56	118.6
C27—C28—C33	117.0 (6)	C55—C56—H56	118.6
C27—C28—C29	125.2 (5)	C56—C57—C58	118.5 (6)
C33—C28—C29	117.8 (5)	C56—C57—H57	120.8
C28—C29—C30	110.0 (5)	C58—C57—H57	120.8
C28—C29—H29A	109.7	C59—C58—C57	121.2 (6)
C30—C29—H29A	109.7	C59—C58—Br1	119.9 (5)
C28—C29—H29B	109.7	C57—C58—Br1	118.9 (5)
C30—C29—H29B	109.7	C60—C59—C58	118.8 (6)
H29A—C29—H29B	108.2	C60—C59—H59	120.6
C29—C30—C31	110.5 (5)	C58—C59—H59	120.6
C29—C30—H30A	109.5	C59—C60—C55	121.7 (5)
C31—C30—H30A	109.5	C59—C60—H60	119.1
C29—C30—H30B	109.5	C55—C60—H60	119.1
C31—C30—H30B	109.5		
C6—C1—C2—C3	1.4 (10)	C29—C28—C33—O3	−177.4 (5)
Br3—C1—C2—C3	−177.1 (5)	C27—C28—C33—C32	−178.8 (5)
C1—C2—C3—C4	−1.5 (10)	C29—C28—C33—C32	4.3 (7)
C2—C3—C4—C5	0.5 (9)	C34—C32—C33—O3	−18.2 (7)
C2—C3—C4—C7	177.9 (6)	C31—C32—C33—O3	162.8 (5)
C3—C4—C5—C6	0.7 (9)	C34—C32—C33—C28	160.0 (5)
C7—C4—C5—C6	−176.5 (6)	C31—C32—C33—C28	−19.0 (7)
C2—C1—C6—C5	−0.2 (10)	C31—C32—C34—C35	1.9 (9)
Br3—C1—C6—C5	178.3 (5)	C33—C32—C34—C35	−176.9 (5)
C4—C5—C6—C1	−0.9 (10)	C32—C34—C35—C40	−179.4 (6)
C5—C4—C7—C8	−27.6 (10)	C32—C34—C35—C36	1.3 (10)
C3—C4—C7—C8	155.1 (6)	C40—C35—C36—C37	2.5 (10)
C4—C7—C8—C13	176.6 (6)	C34—C35—C36—C37	−178.2 (6)
C4—C7—C8—C9	−4.2 (10)	C35—C36—C37—C38	−0.6 (11)
C7—C8—C9—C10	−151.9 (6)	C36—C37—C38—C39	−1.7 (10)
C13—C8—C9—C10	27.4 (8)	C36—C37—C38—Br6	179.9 (5)
C8—C9—C10—C11	−60.5 (7)	C37—C38—C39—C40	1.9 (9)
C9—C10—C11—C12	59.7 (7)	Br6—C38—C39—C40	−179.7 (4)
C10—C11—C12—C14	150.8 (6)	C38—C39—C40—C35	0.2 (9)
C10—C11—C12—C13	−26.3 (8)	C36—C35—C40—C39	−2.3 (9)
C7—C8—C13—O1	3.3 (9)	C34—C35—C40—C39	178.3 (5)
C9—C8—C13—O1	−176.0 (6)	C46—C41—C42—C43	−1.4 (11)
C7—C8—C13—C12	−174.9 (5)	Br2—C41—C42—C43	180.0 (5)
C9—C8—C13—C12	5.8 (9)	C41—C42—C43—C44	0.0 (11)
C14—C12—C13—O1	−2.1 (9)	C42—C43—C44—C45	0.9 (10)
C11—C12—C13—O1	175.2 (6)	C42—C43—C44—C47	179.3 (6)
C14—C12—C13—C8	176.2 (6)	C43—C44—C45—C46	−0.5 (10)

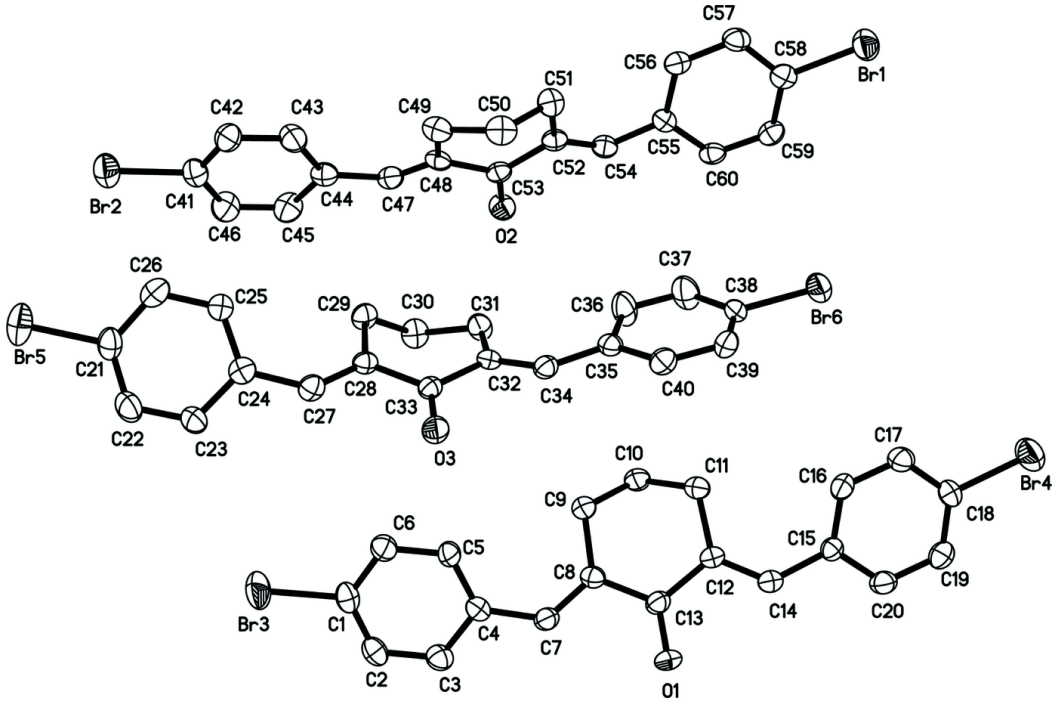
C11—C12—C13—C8	−6.5 (8)	C47—C44—C45—C46	−179.0 (6)
C13—C12—C14—C15	−179.2 (5)	C42—C41—C46—C45	1.8 (11)
C11—C12—C14—C15	3.7 (11)	Br2—C41—C46—C45	−179.6 (5)
C12—C14—C15—C16	27.1 (10)	C44—C45—C46—C41	−0.9 (11)
C12—C14—C15—C20	−153.4 (7)	C43—C44—C47—C48	14.7 (11)
C20—C15—C16—C17	1.8 (9)	C45—C44—C47—C48	−167.0 (7)
C14—C15—C16—C17	−178.6 (6)	C44—C47—C48—C53	179.8 (6)
C15—C16—C17—C18	0.6 (10)	C44—C47—C48—C49	1.2 (11)
C16—C17—C18—C19	−2.6 (10)	C47—C48—C49—C50	163.9 (6)
C16—C17—C18—Br4	179.0 (5)	C53—C48—C49—C50	−14.7 (8)
C17—C18—C19—C20	2.1 (10)	C48—C49—C50—C51	54.0 (7)
Br4—C18—C19—C20	−179.6 (5)	C49—C50—C51—C52	−61.1 (7)
C18—C19—C20—C15	0.4 (10)	C50—C51—C52—C54	−149.0 (6)
C16—C15—C20—C19	−2.4 (9)	C50—C51—C52—C53	28.7 (8)
C14—C15—C20—C19	178.1 (6)	C54—C52—C53—O2	6.9 (8)
C26—C21—C22—C23	−1.2 (12)	C51—C52—C53—O2	−171.0 (6)
Br5—C21—C22—C23	−179.1 (5)	C54—C52—C53—C48	−172.0 (5)
C21—C22—C23—C24	−0.3 (12)	C51—C52—C53—C48	10.1 (8)
C22—C23—C24—C25	1.2 (10)	C47—C48—C53—O2	−15.2 (8)
C22—C23—C24—C27	179.0 (7)	C49—C48—C53—O2	163.4 (6)
C23—C24—C25—C26	−0.7 (10)	C47—C48—C53—C52	163.6 (5)
C27—C24—C25—C26	−178.4 (6)	C49—C48—C53—C52	−17.7 (8)
C22—C21—C26—C25	1.6 (11)	C53—C52—C54—C55	179.7 (6)
Br5—C21—C26—C25	179.6 (5)	C51—C52—C54—C55	−2.5 (10)
C24—C25—C26—C21	−0.7 (10)	C52—C54—C55—C56	−32.2 (10)
C25—C24—C27—C28	−39.4 (10)	C52—C54—C55—C60	149.6 (6)
C23—C24—C27—C28	143.0 (7)	C60—C55—C56—C57	−1.8 (9)
C24—C27—C28—C33	177.2 (5)	C54—C55—C56—C57	−180.0 (6)
C24—C27—C28—C29	−6.1 (10)	C55—C56—C57—C58	−1.2 (10)
C27—C28—C29—C30	−140.6 (6)	C56—C57—C58—C59	2.6 (9)
C33—C28—C29—C30	36.0 (7)	C56—C57—C58—Br1	−179.7 (5)
C28—C29—C30—C31	−63.2 (6)	C57—C58—C59—C60	−0.9 (9)
C29—C30—C31—C32	49.5 (7)	Br1—C58—C59—C60	−178.5 (5)
C30—C31—C32—C34	172.6 (5)	C58—C59—C60—C55	−2.3 (9)
C30—C31—C32—C33	−8.5 (7)	C56—C55—C60—C59	3.6 (9)
C27—C28—C33—O3	−0.5 (8)	C54—C55—C60—C59	−178.1 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C27—H27···O2^{i}	0.93	2.58	3.310 (8)	136
C14—H14···O3^{ii}	0.93	2.59	3.446 (8)	154

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+2, −*y*+1, −*z*.

Fig. 1


C57
C58
Br1
C56
C51
C42
C43
C49
C50
C55
C59
C52
C54
C60
C48
C53
Br2
C41
C44
C47
C46
C45
O2
C26
C37
C38
C25
C29
C30
C31
C36
Br6
Br5
C21
C35
C39
C24
C28
C32
C34
C40
C27
C33
C22
C23
O3
C17
C10
C11
Br4
C16
C9
C18
C6
C5
C15
C8
C12
C19
Br3
C1
C4
C13
C14
C20
C7
C2
C3
O1