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3″-(4-Bromobenzylidene)-4′-(4-bromophenyl)-1′-methylindan-2-spiro-2′-pyrrolidine-3′-spiro-1″-cyclohexane-1,3,2″-trione

B. K. Satis Kumar,[a] D. Gayathri,[b] D. Velmurugan,[b]* K. Ravikumar[c] and M. Poornachandran[d]

[a]Department of Physics, Presidency College, Chennai 600 005, India, [b]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [c]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [d]Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

Correspondence e-mail: d_velu@yahoo.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.047
wR factor = 0.137
Data-to-parameter ratio = 13.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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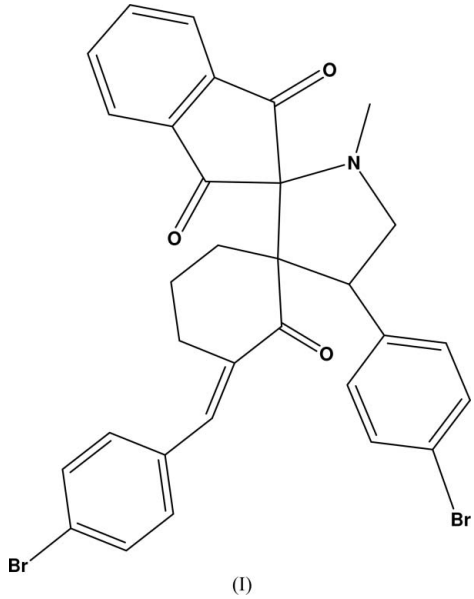
The pyrrolidine ring in the title compound, $C_{31}H_{25}Br_2NO_3$, adopts a twisted conformation and the five-membered ring in the indanedione group adopts a slight envelope conformation. The molecule is stabilized by weak C—H···O intramolecular interactions.

Comment

Heterocyclic compounds have occupied an important place among organic compounds because of their pharmacological properties. Substituted pyrrolidine compounds have attracted attention as they resemble the basic structural elements of many alkaloids and pharmacologically active compounds. Pyrrolidine derivatives are very useful in preventing and treating rheumatoid arthritis, asthma, allergies, rhinitis and related diseases as they inhibit the production of prostaglandin E2 and intracellular phospholipase A2 (Mitsuaki *et al.*, 1997). We present here the crystal structure of the title compound, (I).



(I)

Except at the spiro junctions (Table 1), the bond lengths and angles in the structure of (I) are comparable with literature values (Allen *et al.*, 1987); the deviations are due to bulky substituents. The sum of the bond angles around N1 (343.1°) indicates sp^3 hybridization. Atoms Br1 and Br2 deviate by 0.013 (1) and 0.004 (1) Å, respectively, from the benzene rings to which they are attached. Atoms O2 and O3 deviate by 0.226 (4) and 0.278 (3) Å, respectively, from the plane of atoms C24–C31.

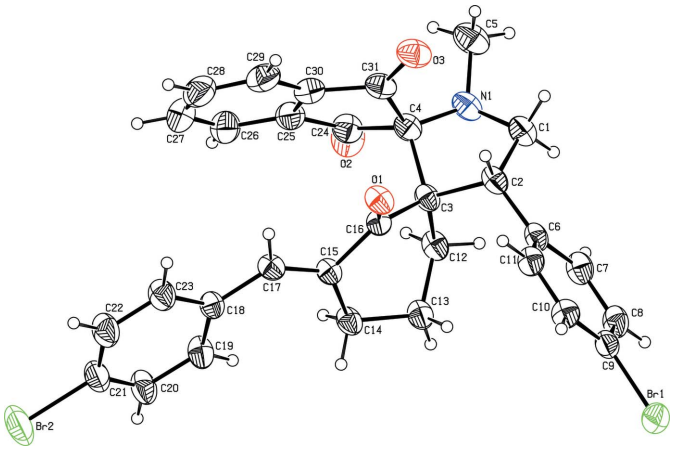


Figure 1
The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

The pyrrolidine ring of (I) adopts a twisted conformation, with a pseudo-twofold rotation axis passing through atom C1 and the mid-point of the C3—C4 bond. The five-membered ring in the indanedione group adopts a slight envelope conformation, with atom C4 deviating by 0.385 (4) Å from the plane of atoms C24/C25/C30/C31 and by 0.436 (4) Å from the plane of atoms C24/C25/C26–C31. The puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameter (Nardelli, 1983) for the pyrrolidine ring are q_2 = 0.472 (4) Å, φ = 118.1 (5)° and $\Delta_2(C_1)$ = 7.3 (4), and the corresponding values for the five-membered ring (C4/C24/C25/C30/C31) are q_2 = 0.237 (5) Å, φ = 360.0 (1)° and $\Delta_s(C_4)$ = 0.2 (4).

The molecule of (I) is stabilized by weak C—H···O intramolecular interactions (Table 2).

Experimental

A mixture of 2,6-bis(*p*-bromobenzylidene)cyclohexanone (1 mmol), ninhydrin (1.2 mmol) and sarcosine (1.2 mmol) in methanol (30 ml) was refluxed until the disappearance of the starting materials, as evidenced by thin-layer chromatography. The solvent was evaporated *in vacuo* and the residue was subjected to column chromatography (silica gel, 100–200 mesh), eluting with hexane–ethyl acetate (9:1 *v*/*v*) to give the title compound, which was recrystallized from methanol.

Crystal data

$C_{31}H_{25}Br_2NO_3$
M_r = 619.34
Monoclinic, $P2_1/c$
a = 14.3345 (10) Å
b = 13.0061 (9) Å
c = 14.2616 (10) Å
β = 99.223 (1)°
V = 2624.5 (3) Å^3
Z = 4
D_x = 1.567 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 3.12 mm^{-1}
T = 293 (2) K
Block, colourless
0.25 × 0.24 × 0.22 mm

Data collection

Bruker SMART APEX diffractometer
ω scans
Absorption correction: none
24763 measured reflections
4613 independent reflections
3382 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.137
S = 1.01
4613 reflections
335 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0691P)^2 + 2.4563P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—C3	1.573 (5)	C4—N1	1.434 (5)
C3—C16	1.530 (5)	C4—C24	1.548 (6)
C3—C12	1.540 (5)	C4—C31	1.554 (6)
C3—C4	1.582 (5)		
C16—C3—C12	112.0 (3)	N1—C4—C24	115.5 (3)
C16—C3—C2	111.9 (3)	N1—C4—C31	117.8 (3)
C12—C3—C2	111.5 (3)	C24—C4—C31	100.1 (3)
C16—C3—C4	110.7 (3)	N1—C4—C3	101.2 (3)
C12—C3—C4	111.9 (3)	C24—C4—C3	112.0 (3)
C2—C3—C4	98.0 (3)	C31—C4—C3	110.7 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1	0.98	2.47	2.855 (5)	103
C2—H2···O3	0.98	2.57	3.190 (5)	122
C12—H12A···O2	0.97	2.41	3.196 (5)	138
C17—H17···O1	0.93	2.40	2.778 (5)	104

H atoms were positioned geometrically and allowed to ride on their parent C atoms, with C—H distances in the range 0.93–0.98 Å and with U_{iso}(H) = 1.2 U_{eq}(C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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