Refinement

Refinement on F
$R = 0.031$
$wR = 0.038$
$S = 1.06$
1219 reflections
190 parameters
$w = 1/[\sigma^2(F) + 0.0030|F|^2]$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: none
Atomic scattering factors
from *SHELXTL-Plus*
(Sheldrick, 1991)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Br	0.5686 (1)	0.0535 (1)	0.61914 (4)	0.0649 (2)
O(1)	0.8543 (5)	−0.0620 (3)	0.7315 (3)	0.080 (2)
O(2)	1.4351 (5)	−0.1572 (3)	0.8333 (3)	0.092 (2)
O(3)	1.5903 (6)	−0.0533 (3)	0.9205 (4)	0.111 (2)
O(4)	1.5623 (4)	0.1473 (2)	0.8791 (2)	0.052 (1)
N(1)	1.4684 (6)	−0.0692 (3)	0.8641 (3)	0.057 (2)
C(1)	0.7723 (6)	0.1143 (4)	0.6856 (3)	0.051 (2)
C(2)	0.8944 (7)	0.0309 (4)	0.7294 (3)	0.047 (2)
C(3)	1.0660 (6)	0.0679 (3)	0.7694 (3)	0.040 (2)
C(4)	1.1147 (6)	0.1738 (4)	0.7791 (3)	0.045 (2)
C(5)	1.2775 (6)	0.2019 (4)	0.8164 (3)	0.045 (2)
C(6)	1.3989 (6)	0.1252 (4)	0.8454 (3)	0.043 (2)
C(7)	1.3503 (6)	0.0174 (3)	0.8357 (3)	0.042 (2)
C(8)	1.1876 (6)	−0.0096 (4)	0.7985 (3)	0.046 (2)
C(9)	1.6056 (6)	0.2548 (3)	0.9049 (3)	0.051 (2)
C(10)	1.7903 (6)	0.2565 (4)	0.9471 (3)	0.046 (2)
C(11)	1.8861 (6)	0.3481 (4)	0.9442 (3)	0.054 (2)
C(12)	2.0522 (7)	0.3522 (5)	0.9863 (4)	0.063 (2)
C(13)	2.1257 (7)	0.2650 (5)	1.0325 (4)	0.065 (2)
C(14)	2.0314 (6)	0.1726 (5)	1.0348 (4)	0.061 (2)
C(15)	1.8655 (6)	0.1675 (4)	0.9925 (3)	0.053 (2)

Table 2. *Selected geometric parameters* (Å, °)

Br—C(1)	1.926 (5)	O(1)—C(2)	1.214 (6)
O(2)—N(1)	1.211 (6)	O(3)—N(1)	1.197 (6)
O(4)—C(6)	1.350 (5)	O(4)—C(9)	1.435 (5)
N(1)—C(7)	1.461 (6)	C(1)—C(2)	1.511 (7)
C(2)—C(3)	1.481 (7)	C(3)—C(4)	1.391 (6)
C(3)—C(8)	1.397 (7)	C(4)—C(5)	1.377 (6)
C(5)—C(6)	1.389 (6)	C(6)—C(7)	1.413 (6)
C(7)—C(8)	1.372 (7)	C(9)—C(10)	1.509 (7)
C(10)—C(11)	1.382 (7)	C(10)—C(15)	1.393 (7)
C(11)—C(12)	1.377 (7)	C(12)—C(13)	1.375 (8)
C(13)—C(14)	1.383 (8)	C(14)—C(15)	1.377 (7)
Br—C(1)—C(2)	112.5 (3)	C(6)—O(4)—C(9)	118.4 (3)
O(2)—N(1)—O(3)	121.4 (5)	O(3)—N(1)—C(7)	119.9 (4)
O(2)—N(1)—C(7)	118.6 (4)	O(1)—C(2)—C(1)	121.7 (5)
C(1)—C(2)—C(3)	116.7 (4)	O(1)—C(2)—C(3)	121.5 (4)
C(2)—C(3)—C(8)	117.3 (4)	C(2)—C(3)—C(4)	124.7 (4)
C(4)—C(3)—C(8)	118.0 (4)	C(3)—C(4)—C(5)	121.3 (4)
C(4)—C(5)—C(6)	120.9 (4)	O(4)—C(6)—C(5)	123.9 (4)
C(5)—C(6)—C(7)	118.0 (4)	O(4)—C(6)—C(7)	118.0 (4)
N(1)—C(7)—C(6)	122.2 (4)	C(6)—C(7)—C(8)	120.5 (4)
N(1)—C(7)—C(8)	117.3 (4)	C(3)—C(8)—C(7)	121.3 (4)
O(4)—C(9)—C(10)	107.8 (4)	C(9)—C(10)—C(15)	120.9 (4)
C(9)—C(10)—C(11)	120.3 (4)	C(11)—C(10)—C(15)	118.8 (5)
C(10)—C(11)—C(12)	120.9 (5)	C(11)—C(12)—C(13)	120.4 (5)
C(12)—C(13)—C(14)	119.1 (15)	C(13)—C(14)—C(15)	120.8 (5)
C(10)—C(15)—C(14)	119.9 (5)		

The $\Delta\rho$ map showed the positions of all H atoms. H atoms were placed in idealized positions and included in the least-squares refinement with fixed isotropic temperature factors. Structure solution and refinement were performed with *SHELXTL-Plus* (Sheldrick, 1991). Geometrical parameters were calculated using the program *PARST* (Nardelli, 1983).

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2-Methyl-2-(4-nitrophenylazo)-1,3-indandione, C$_{16}$H$_{11}$N$_3$O$_4$

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Abstract

The 2-methyl-1,3-indandione and 4-nitrophenyl groups are *trans* to each other. The five-membered ring of the indandione moiety adopts an envelope

conformation. The phenyl and azo groups are not coplanar because of steric hindrance between the lone-pair orbitals at the carbonyl O and β-N atoms [C1'—N=N = 112.6 (6) and 112.8 (6)° for molecules A and B, respectively].

Comment

The structure determination of the title compound (I) was undertaken in order to establish the configuration at the azo group.

(I)
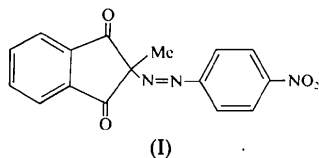
The 2-methyl-1,3-indandione and the 4-nitro-phenyl groups are *trans*, as expected. Each five-membered ring of the two independent molecules in the asymmetric unit has an envelope conformation with C2 as the out-of-plane atom. For the molecules A and B the angles between the least-squares plane through atoms C3, C4, C9 and C1, and the plane of C3, C2 and C1 are 9.2 (5) and 11.3 (5)°, respectively.

In the phenylazo groups of the two independent molecules, the benzene ring plane and the azo group deviate slightly from coplanarity [N2—N1—C1' —C6' = 4.4 (7) and -171.8 (7)° for molecules A and B, respectively, and N2—N1—C1'—C2' = 174.0 (7) and 11.1 (7)° for molecules A and B, respectively]. The NO$_2$ plane is slightly twisted out of the plane of the phenyl ring [by 3.5 (5) and 6.3 (5)° for molecules A and B, respectively] in order to minimize the steric hindrance between the O atoms and both H3' and H5', but still allowing some π conjugation with the phenyl ring. The close O4···H3' and O3···H5' contacts of 2.47 (1) and 2.41 (1) Å, respectively, for molecule A [2.40 (1) and 2.44 (1) Å, respectively, for molecule B] are a result of this compromise.
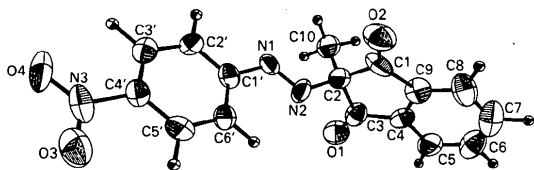

Fig. 1. A view of the molecule showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels.

Experimental

Crystal data

$C_{16}H_{11}N_3O_4$
$M_r = 309.28$
Orthorhombic
$P2_12_12_1$
$a = 6.890$ (2) Å
$b = 15.798$ (4) Å
$c = 27.201$ (7) Å
$V = 2961$ (1) Å^3
$Z = 8$
$D_x = 1.387$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 15 reflections
$\theta = 6$–20°
$\mu = 0.096$ mm^{-1}
$T = 293$ K
Plate
0.3 × 0.3 × 0.2 mm
Yellow

Data collection

Nicolet $R3m/V$ diffractometer
ω–2θ scans
Absorption correction: empirical
$T_{min} = 0.914$, $T_{max} = 0.924$
1205 measured reflections
1205 independent reflections

1088 observed reflections
[$F > 4\sigma(F)$]
$\theta_{max} = 22.5°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 28$
2 standard reflections
monitored every 125 reflections
intensity variation: 1%

Refinement

Refinement on F
$R = 0.043$
$wR = 0.039$
$S = 1.5$
967 reflections
420 parameters
Only coordinates of H atoms refined

$w = 0.8515/[\sigma^2(F) + 0.00054F^2]$
$(\Delta/\sigma)_{max} = 0.05$
$\Delta\rho_{max} = 0.145$ e Å^{-3}
$\Delta\rho_{min} = -0.142$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1A	0.5278 (11)	0.0648 (4)	0.2419 (2)	0.077 (3)
O2A	1.1226 (11)	0.1742 (4)	0.2999 (2)	0.081 (3)
O3A	0.9985 (13)	−0.2500 (5)	0.4910 (3)	0.106 (3)
O4A	0.9593 (10)	−0.1610 (4)	0.5500 (2)	0.107 (3)
N1A	0.7908 (10)	0.0777 (4)	0.3687 (2)	0.047 (3)
N2A	0.8097 (10)	0.0572 (4)	0.3247 (2)	0.056 (3)
N3A	0.9568 (13)	−0.1786 (6)	0.5070 (3)	0.076 (4)
C4'A	0.9088 (12)	−0.1135 (6)	0.4707 (3)	0.057 (4)
C3'A	0.8690 (12)	−0.0313 (6)	0.4864 (3)	0.051 (4)
C2'A	0.8280 (11)	0.0289 (5)	0.4517 (3)	0.052 (4)
C1'A	0.8285 (12)	0.0077 (5)	0.4022 (3)	0.045 (3)
C6'A	0.8673 (12)	−0.0745 (6)	0.3866 (3)	0.048 (4)
C5'A	0.9044 (11)	−0.1352 (5)	0.4205 (3)	0.050 (4)
C10A	0.6762 (15)	0.2072 (5)	0.3119 (3)	0.076 (4)
C2A	0.7851 (16)	0.1293 (5)	0.2900 (3)	0.057 (4)
C3A	0.6832 (16)	0.0957 (6)	0.2438 (3)	0.058 (4)
C4A	0.8165 (16)	0.1145 (5)	0.2026 (3)	0.051 (4)
C9A	0.9938 (17)	0.1477 (6)	0.2188 (3)	0.061 (4)
C1A	0.9912 (17)	0.1537 (6)	0.2736 (4)	0.065 (5)
C8A	1.1489 (16)	0.1647 (6)	0.1870 (4)	0.079 (5)
C7A	1.1147 (18)	0.1495 (7)	0.1378 (5)	0.093 (5)
C6A	0.9386 (18)	0.1186 (7)	0.1208 (4)	0.082 (5)
C5A	0.7906 (17)	0.0991 (5)	0.1524 (4)	0.072 (5)
O1B	0.6755 (10)	0.1608 (4)	0.5480 (3)	0.097 (3)
O2B	0.0879 (10)	0.0383 (5)	0.6014 (2)	0.086 (3)
O3B	0.4008 (12)	−0.1302 (5)	0.2798 (3)	0.145 (4)
O4B	0.4615 (14)	−0.2255 (5)	0.3299 (2)	0.120 (4)
N1B	0.3103 (10)	0.0757 (4)	0.4760 (3)	0.064 (3)
N2B	0.3540 (11)	0.0503 (4)	0.5171 (3)	0.069 (3)
N3B	0.4122 (13)	−0.1549 (5)	0.3205 (3)	0.077 (4)
C4'B	0.3821 (12)	−0.0963 (6)	0.3618 (3)	0.048 (3)

C3'B	0.4001 (12)	−0.1273 (5)	0.4095 (3)	0.049 (4)	C8A—C7A—C6A	122.3 (11)	C8B—C7B—C6B 124.4 (10)
C2'B	0.3740 (12)	−0.0729 (6)	0.4481 (3)	0.054 (4)	C7A—C6A—C5A	121.4 (11)	C7B—C6B—C5B 120.8 (11)
C1'B	0.3371 (11)	0.0123 (5)	0.4384 (3)	0.045 (3)	C4A—C5A—C6A	118.6 (10)	C4B—C5B—C6B 117.0 (10)
C6'B	0.3143 (12)	0.0417 (6)	0.3904 (3)	0.058 (4)			
C5'B	0.3404 (13)	−0.0133 (5)	0.3517 (3)	0.056 (4)			
C10B	0.2256 (15)	0.1960 (5)	0.5422 (4)	0.080 (5)			
C2B	0.3339 (16)	0.1159 (5)	0.5562 (3)	0.057 (4)			
C3B	0.5383 (17)	0.1362 (6)	0.5730 (4)	0.063 (4)			
C4B	0.5502 (16)	0.1235 (6)	0.6278 (4)	0.057 (4)			
C9B	0.3807 (16)	0.0851 (5)	0.6427 (3)	0.049 (4)			
C1B	0.2466 (16)	0.0735 (7)	0.6008 (4)	0.064 (4)			
C8B	0.3442 (15)	0.0629 (6)	0.6922 (3)	0.067 (4)			
C7B	0.4990 (18)	0.0805 (7)	0.7236 (4)	0.082 (4)			
C6B	0.6672 (17)	0.1174 (7)	0.7094 (4)	0.089 (5)			
C5B	0.7008 (14)	0.1371 (6)	0.6602 (4)	0.084 (5)			

Empirical absorption corrections based on an ellipsoidal fit to ψ-scan data were applied (Kopfmann & Huber, 1968). The H atoms of the benzene rings were refined riding on the parent C atoms at a distance of 1.08 Å. Each Me group was refined as a rigid group. The H atoms had fixed isotropic temperature factors. Refinement was by blocked full-matrix least-squares methods. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985); *SIR*88 (Burla *et al.*, 1989). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983). Molecular graphics: *ORTEP*II (Johnson, 1976).

Table 2. *Selected geometric parameters* (Å, °)

O1A—C3A	1.18 (1)	O1B—C3B	1.23 (1)
O2A—C1A	1.20 (1)	O2B—C1B	1.23 (1)
O3A—N3A	1.24 (1)	O3B—N3B	1.18 (1)
O4A—N3A	1.20 (1)	O4B—N3B	1.19 (1)
N1A—N2A	1.25 (1)	N1B—N2B	1.23 (1)
N1A—C1'A	1.46 (1)	N1B—C1'B	1.44 (1)
N2A—C2A	1.49 (1)	N2B—C2B	1.49 (1)
N3A—C4'A	1.47 (1)	N3B—C4'B	1.47 (1)
C4'A—C3'A	1.39 (1)	C4'B—C3'B	1.39 (1)
C4'A—C5'A	1.41 (1)	C4'B—C5'B	1.37 (1)
C3'A—C2'A	1.37 (1)	C3'B—C2'B	1.37 (1)
C2'A—C1'A	1.39 (1)	C2'B—C1'B	1.40 (1)
C1'A—C6'A	1.39 (1)	C1'B—C6'B	1.40 (1)
C6'A—C5'A	1.36 (1)	C6'B—C5'B	1.38 (1)
C10A—C2A	1.56 (1)	C10B—C2B	1.52 (1)
C2A—C3A	1.53 (1)	C2B—C3B	1.52 (1)
C2A—C1A	1.54 (2)	C2B—C1B	1.51 (1)
C3A—C4A	1.48 (1)	C3B—C4B	1.51 (1)
C4A—C9A	1.40 (2)	C4B—C9B	1.38 (2)
C4A—C5A	1.40 (1)	C4B—C5B	1.38 (2)
C9A—C1A	1.49 (1)	C9B—C1B	1.48 (1)
C9A—C8A	1.40 (2)	C9B—C8B	1.41 (1)
C8A—C7A	1.38 (2)	C8B—C7B	1.39 (2)
C7A—C6A	1.39 (2)	C7B—C6B	1.35 (2)
C6A—C5A	1.37 (2)	C6B—C5B	1.39 (2)
N2A—N1A—C1'A	112.6 (6)	N2B—N1B—C1'B	112.8 (6)
N1A—N2A—C2A	113.4 (6)	N1B—N2B—C2B	113.6 (6)
O3A—N3A—O4A	123.4 (9)	O3B—N3B—O4B	122.0 (9)
O4A—N3A—C4'A	119.6 (9)	O4B—N3B—C4'B	117.7 (8)
O3A—N3A—C4'A	117.0 (8)	O3B—N3B—C4'B	120.0 (9)
N3A—C4'A—C5'A	119.2 (8)	N3B—C4'B—C5'B	118.6 (7)
N3A—C4'A—C3'A	119.5 (7)	N3B—C4'B—C3'B	118.5 (8)
C3'A—C4'A—C5'A	121.3 (8)	C3'B—C4'B—C5'B	122.8 (8)
C4'A—C3'A—C2'A	118.4 (8)	C4'B—C3'B—C2'B	118.8 (7)
C3'A—C2'A—C1'A	120.1 (8)	C3'B—C2'B—C1'B	119.0 (7)
N1A—C1'A—C2'A	115.1 (7)	N1B—C1'B—C2'B	123.9 (7)
C2'A—C1'A—C6'A	121.4 (8)	C2'B—C1'B—C6'B	121.2 (7)
N1A—C1'A—C6'A	123.5 (7)	N1B—C1'B—C6'B	114.7 (7)
C1'A—C6'A—C5'A	119.3 (8)	C1'B—C6'B—C5'B	119.4 (8)
C4'A—C5'A—C6'A	119.4 (8)	C4'B—C5'B—C6'B	118.5 (8)
N2A—C2A—C10A	114.6 (7)	N2B—C2B—C10B	116.6 (7)
C10A—C2A—C1A	110.9 (7)	C10B—C2B—C1B	112.0 (8)
C10A—C2A—C3A	111.5 (7)	C10B—C2B—C3B	110.9 (8)
N2A—C2A—C1A	105.7 (7)	N2B—C2B—C1B	107.5 (7)
N2A—C2A—C3A	107.9 (7)	N2B—C2B—C3B	106.0 (8)
C3A—C2A—C1A	105.8 (7)	C3B—C2B—C1B	102.8 (7)
O1A—C3A—C2A	126.5 (8)	O1B—C3B—C2B	128.0 (9)
C2A—C3A—C4A	105.5 (7)	C2B—C3B—C4B	108.7 (8)
O1A—C3A—C4A	127.9 (8)	O1B—C3B—C4B	123.3 (9)
C3A—C4A—C5A	128.7 (9)	C3B—C4B—C5B	130.8 (9)
C3A—C4A—C9A	112.2 (7)	C3B—C4B—C9B	107.7 (9)
C9A—C4A—C5A	118.9 (9)	C9B—C4B—C5B	121.3 (8)
C4A—C9A—C8A	122.8 (8)	C4B—C9B—C8B	122.7 (8)
C4A—C9A—C1A	109.1 (9)	C4B—C9B—C1B	111.0 (8)
C1A—C9A—C8A	127.8 (9)	C1B—C9B—C8B	126.3 (9)
C2A—C1A—C9A	106.5 (9)	C2B—C1B—C9B	108.4 (8)
O2A—C1A—C9A	127.1 (10)	O2B—C1B—C9B	127.1 (8)
O2A—C1A—C2A	126.3 (9)	O2B—C1B—C2B	124.5 (8)
C9A—C8A—C7A	115.8 (9)	C9B—C8B—C7B	113.4 (9)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond angles involving H atoms and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71644 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1042]

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Butyl[3-(1,1,3,3,5,5,5-heptamethyltrisiloxan-1-yl)propyl]dimethylammonium Bromide, $C_{16}H_{42}NO_2Si_3^+.Br^-$, at 173 and 301 K

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Abstract

The molecules form double layers in head–head arrangements. The non-polar regions of the layers are held together by weak van der Waals interactions and the polar regions mainly by electrostatic interactions. One of the Si—O—Si bond angles is much