weighting scheme was $w^{-1} = 1 + 0{\cdot}0025(F_o - 18{\cdot}5)^2$ for $F_o > 18{\cdot}5$ and $w^{-1} = 1 + 0{\cdot}004(18{\cdot}5 - F_o)^2$ for $F_o < 18{\cdot}5$. Final value of isotropic extinction parameter was $g = 5{\cdot}6\ (16) \times 10^{-8}$ where $F_c' = F_c(1 - gF_c^2/\sin\theta)$. Maximum range on $\Delta\rho$ map $\pm 0{\cdot}19$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations performed using an Acorn ACW443 workstation.

Final atomic parameters (excluding H atoms) are listed in Table 1 and selected bond lengths and angles are listed in Table 2.* Fig. 1 shows the atomic labelling scheme.

Discussion. The obvious and expected difference between this determination (at low temperature) and the earlier room-temperature study is a sharp reduction in the magnitude of the atomic thermal parameters, which is especially pronounced for the aliphatic end chains. Associated with this is an improvement in the e.s.d.'s of the atomic coordinates. Our coordinates and atom labels have been transformed to correspond to those of the earlier study.

Lowering the temperature makes little difference to the molecular geometry. Torsion angles, bond angles and bond lengths are essentially unchanged, with the exception of a slight enhancement in regularity of bond lengths within the benzene units. The low-temperature structure retains the asymmetry of bond angles at the substituent-bearing carbons of the benzene rings and confirms the banana shape of the central hexadiyne system. The average C≡C—C angle in the hexadiyne unit is 176·9°, and the distortions from linearity are all in the same sense. These are distortions that we would tentatively associate with the type of competition between alternative molecular packing requirements that were described in the *Introduction*. The molecular packing at low temperature is the same as that at room temperature except for rather small changes to intermolecular distances.

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* Lists of structure factors, anisotropic thermal parameters for C and O atoms, and fractional coordinates and isotropic temperature factors for H atoms have been deposited with British Library Document Supply Centre as Supplementary Publication No. SUP 51499 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1-Methyl-3,3′-dioxo-2′-phenylspiro[indole-2,1′-isoindole], a Novel Rearrangement Product from Reaction of Aniline with a Dioxodibenz[*b,f*]azocine

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(*Received* 3 *May* 1988; *accepted* 18 *October* 1988)

Abstract. $C_{22}H_{16}N_2O_2$, $M_r = 340{\cdot}39$, monoclinic, $P2_1/c$, $a = 14{\cdot}096\ (2)$, $b = 13{\cdot}891\ (2)$, $c = 9{\cdot}022\ (2)$ Å, $\beta = 103{\cdot}03\ (1)°$, $V = 1721\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}313$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7170 Å, $\mu = 0{\cdot}796$ cm^{-1}, $F(000) = 712$, $T = 294$ K, $R = 0{\cdot}034$ for 1253 unique reflections with $|F_o| > 3\sigma(|F_o|)$. The present study established the structure of the title compound obtained from a temperature-dependent rearrangement reactions have been found to occur (Letcher & occurs with the two planes at right angles (90·1°) to each other and with no unusual intramolecular distances or angles.

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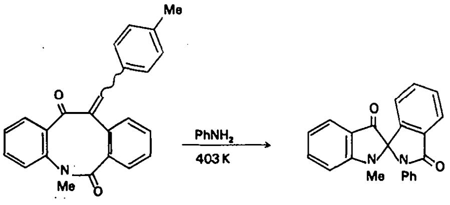
Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O(1)	0·3796 (2)	0·0308 (2)	0·3584 (2)	6·08 (6)
O(2)	0·0802 (2)	0·1428 (2)	0·1431 (3)	6·32 (6)
N(1)	0·2457 (2)	−0·1356 (2)	0·1006 (2)	3·83 (5)
N(2)	0·1749 (2)	0·0065 (2)	0·1905 (2)	3·64 (5)
C(1)	0·2592 (2)	−0·0354 (2)	0·1464 (3)	3·47 (6)
C(2)	0·3512 (2)	−0·0388 (2)	0·2790 (3)	4·00 (7)
C(3)	0·3900 (2)	−0·1348 (2)	0·2798 (3)	4·02 (7)
C(4)	0·4755 (2)	−0·1753 (3)	0·3688 (4)	5·80 (8)
C(5)	0·4952 (3)	−0·2693 (3)	0·3400 (4)	7·17 (9)
C(6)	0·4323 (2)	−0·3223 (2)	0·2290 (4)	6·87 (9)
C(7)	0·3464 (2)	−0·2840 (2)	0·1425 (4)	5·39 (8)
C(8)	0·3260 (2)	−0·1879 (2)	0·1698 (3)	3·91 (6)
C(9)	0·1456 (2)	0·0921 (2)	0·1190 (3)	4·37 (7)
C(10)	0·2740 (2)	0·0332 (2)	0·0234 (3)	3·94 (7)
C(11)	0·3415 (2)	0·0274 (2)	−0·0669 (3)	5·20 (8)
C(12)	0·3407 (2)	0·1019 (3)	−0·1701 (4)	6·49 (9)
C(13)	0·2758 (3)	0·1781 (3)	−0·1812 (4)	7·0 (1)
C(14)	0·2083 (3)	0·1825 (3)	−0·0916 (4)	6·04 (9)
C(15)	0·2089 (2)	0·1078 (2)	0·0116 (3)	4·29 (7)
C(16)	0·1352 (2)	−0·0325 (2)	0·3112 (3)	3·59 (6)
C(17)	0·1709 (2)	−0·0009 (2)	0·4585 (3)	4·39 (7)
C(18)	0·1329 (2)	−0·0390 (3)	0·5749 (3)	5·11 (8)
C(19)	0·0610 (2)	−0·1075 (2)	0·5447 (3)	5·03 (8)
C(20)	0·0255 (2)	−0·1387 (2)	0·3980 (4)	4·91 (8)
C(21)	0·0625 (2)	−0·1012 (2)	0·2789 (3)	4·40 (7)
C(22)	0·1826 (2)	−0·1614 (2)	−0·0448 (4)	5·34 (8)

Introduction. With hydroxylamine, and with primary aliphatic amines, 5-methyl-11-[(4-methylphenyl)-methylene]-6,12-dioxodibenz[*b*,*f*]azocine (1) gives oxaza- and diazachrysene derivatives, *via* rearrangement reactions which probably involve transannular interactions (Letcher, Ng & Cheung, 1987). With aromatic amines, completely different rearrangement reactions have been found to occur (Letcher & Ng, 1989), *e.g.* aniline and (1) at 403 K gave (2). The structural assignment (2) was made from spectral analysis, especially ^{1}H and ^{13}C NMR, and we now report the confirmation of this novel structure, from a single-crystal X-ray study.



(1) (2)

Experimental. The title compound, prepared in 73% yield from the reaction between (1) and aniline at 403 K, was crystallized from chloroform–light petroleum (b.p. 40–60°).

Pale-yellow crystal of dimensions 0·2 × 0·4 × 0·4 mm. Enraf–Nonius CAD-4 diffractometer, with graphite-monochromated Mo *Kα* radiation, and

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

O(1)—C(2)	1·216 (4)	C(6)—C(7)	1·389 (4)
O(2)—C(9)	1·218 (4)	C(7)—C(8)	1·400 (4)
N(1)—C(1)	1·453 (3)	C(9)—C(15)	1·475 (5)
N(1)—C(8)	1·372 (3)	C(10)—C(11)	1·388 (5)
N(1)—C(22)	1·454 (3)	C(10)—C(15)	1·372 (4)
N(2)—C(1)	1·457 (4)	C(11)—C(12)	1·392 (5)
N(2)—C(9)	1·371 (4)	C(12)—C(13)	1·387 (5)
N(2)—C(16)	1·438 (4)	C(13)—C(14)	1·383 (6)
C(1)—C(2)	1·554 (3)	C(14)—C(15)	1·392 (5)
C(1)—C(10)	1·512 (4)	C(16)—C(17)	1·382 (4)
C(2)—C(3)	1·441 (4)	C(16)—C(21)	1·382 (4)
C(3)—C(4)	1·407 (4)	C(17)—C(18)	1·386 (5)
C(3)—C(8)	1·392 (4)	C(18)—C(19)	1·372 (4)
C(4)—C(5)	1·373 (5)	C(19)—C(20)	1·375 (4)
C(5)—C(6)	1·390 (5)	C(20)—C(21)	1·397 (5)

C(1)—N(1)—C(8)	109·6 (2)	N(1)—C(8)—C(7)	127·3 (2)
C(1)—N(1)—C(22)	120·5 (3)	C(3)—C(8)—C(7)	120·4 (2)
C(8)—N(1)—C(22)	123·8 (2)	O(2)—C(9)—N(2)	125·2 (3)
C(1)—N(2)—C(9)	113·3 (2)	O(2)—C(9)—C(15)	128·8 (3)
C(1)—N(2)—C(16)	122·4 (3)	N(2)—C(9)—C(15)	105·9 (2)
C(9)—N(2)—C(16)	123·8 (2)	C(1)—C(10)—C(11)	128·4 (3)
N(1)—C(1)—N(2)	113·5 (2)	C(1)—C(10)—C(15)	109·5 (3)
N(1)—C(1)—C(2)	103·0 (3)	C(11)—C(10)—C(15)	122·2 (3)
N(1)—C(1)—C(10)	115·2 (2)	C(10)—C(11)—C(12)	116·6 (3)
N(2)—C(1)—C(2)	113·1 (2)	C(11)—C(12)—C(13)	121·4 (3)
N(2)—C(1)—C(10)	101·8 (3)	C(12)—C(13)—C(14)	121·4 (3)
C(2)—C(1)—C(10)	110·8 (2)	C(13)—C(14)—C(15)	117·2 (3)
O(1)—C(2)—C(1)	122·5 (2)	C(9)—C(15)—C(10)	109·3 (3)
O(1)—C(2)—C(3)	131·2 (2)	C(9)—C(15)—C(14)	129·5 (3)
C(1)—C(2)—C(3)	106·4 (2)	C(10)—C(15)—C(14)	121·3 (4)
C(2)—C(3)—C(4)	130·4 (3)	N(2)—C(16)—C(17)	119·3 (2)
C(2)—C(3)—C(8)	107·6 (2)	N(2)—C(16)—C(21)	119·8 (2)
C(4)—C(3)—C(8)	121·9 (3)	C(17)—C(16)—C(21)	120·9 (3)
C(3)—C(4)—C(5)	117·2 (3)	C(16)—C(17)—C(18)	119·2 (3)
C(4)—C(5)—C(6)	121·1 (3)	C(17)—C(18)—C(19)	120·7 (3)
C(5)—C(6)—C(7)	122·4 (3)	C(18)—C(19)—C(20)	120·1 (4)
C(6)—C(7)—C(8)	117·0 (3)	C(19)—C(20)—C(21)	120·3 (3)
N(1)—C(8)—C(3)	112·3 (2)	C(16)—C(21)—C(20)	118·9 (3)

C(8)—N(1)—C(1)—N(2)	−133·4 (2)	N(1)—C(1)—C(2)—O(1)	−172·0 (3)
C(8)—N(1)—C(1)—C(2)	−10·9 (3)	N(1)—C(1)—C(2)—C(3)	9·1 (3)
C(8)—N(1)—C(1)—C(10)	109·8 (3)	N(2)—C(1)—C(2)—O(1)	−49·2 (4)
C(22)—N(1)—C(1)—N(2)	73·2 (3)	N(2)—C(1)—C(2)—C(3)	132·0 (2)
C(22)—N(1)—C(1)—C(2)	−164·3 (2)	C(10)—C(1)—C(2)—O(1)	64·4 (3)
C(22)—N(1)—C(1)—C(10)	−43·6 (3)	C(10)—C(1)—C(2)—C(3)	−114·5 (3)
C(9)—N(2)—C(1)—N(1)	−129·3 (2)	N(1)—C(1)—C(10)—C(11)	−53·4 (4)
C(9)—N(2)—C(1)—C(2)	114·0 (3)	N(1)—C(1)—C(10)—C(15)	127·4 (3)
C(9)—N(2)—C(1)—C(10)	−4·9 (3)	N(2)—C(1)—C(10)—C(11)	−176·6 (3)
C(16)—N(2)—C(1)—N(1)	58·2 (3)	N(2)—C(1)—C(10)—C(15)	4·2 (3)
C(16)—N(2)—C(1)—C(2)	−58·5 (3)	C(2)—C(1)—C(10)—C(11)	62·9 (4)
C(16)—N(2)—C(1)—C(10)	−177·4 (2)	C(2)—C(1)—C(10)—C(15)	−116·3 (3)

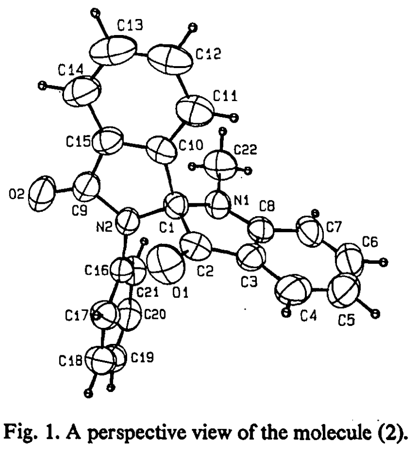


Fig. 1. A perspective view of the molecule (2).

scintillation counter. Cell parameters by least squares from setting angles of 25 reflections with $9 < 2\theta < 13°$ measured on the diffractometer. The intensities of 4342 reflections, $2\theta_{\max} = 46°$, in the range $-15 \leq h \leq 15$, $0 \leq k \leq 15$, $-9 \leq l \leq 9$, were measured using the ω–2θ scan, ω-scan angle $(0{\cdot}60 + 0{\cdot}344\tan\theta)°$ at 0·92–5·49° min^{-1}, extended 25% on each side for background measurement. With a low decay of 0·3% in the three standard reflections and a low absorption coefficient, the data were only corrected for Lorentz and polarization effects. After symmetry-equivalent reflections were averaged, 2209 reflections were obtained, $R_{\rm int} = 0{\cdot}023$; 1253 reflections with $I > 1{\cdot}5\sigma(I)$, where $\sigma^2(I) = S + 4(B1 + B2)$, S = scan, $B1$ and $B2$ = background counts, were considered observed. The structure was determined by direct methods (*MULTAN*11/82: Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least squares minimizing $\sum w|F_o - F_c|^2$ on 235 variables, C, N and O refined anisotropically, H atoms in calculated positions were not refined,* $w = 4F_o^2/[\sigma^2(F_o)^2 + (0{\cdot}04F_o^2)^2]$, atomic scattering factors from *International Tables for X-ray Crystallography* (1974) using the *SDP* programs (Enraf–Nonius, 1985) on a MicroVAX II computer. Final $R = 0{\cdot}034$, $wR = 0{\cdot}042$ and $S = 1{\cdot}296$ for the 1253 reflections. $(\Delta/\sigma)_{\max} = 0{\cdot}01$. A final difference Fourier map was featureless with maximum positive and negative peaks both of 0·14 e Å^{-3}.

Discussion. The *ORTEP* drawing (Johnson, 1976) of the molecule with atom-numbering scheme is shown in Fig. 1. The atomic parameters of the C, N and O atoms are listed in Table 1. Bond lengths, bond angles, and selected torsion angles are given in Table 2.

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51516 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 3-(2-Methylaminophenyl)-2-phenyl-1-isoquinolone, from a Novel Rearrangement of a Dioxodibenz[*b*,*f*]azocine and Aniline

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(*Received* 6 *May* 1988; *accepted* 18 *October* 1988)

Abstract. $C_{22}H_{18}N_2O$, $M_r = 327{\cdot}41$, monoclinic, $P2_1/c$, $a = 15{\cdot}588\,(2)$, $b = 14{\cdot}635\,(2)$, $c = 7{\cdot}966\,(3)$ Å, $\beta = 103{\cdot}87\,(1)°$, $V = 1764\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}232$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}7170$ Å, $\mu = 0{\cdot}711$ cm^{-1}, $F(000) = 688$, $T = 296$ K, $R = 0{\cdot}041$ for 1394 unique reflections with $|F_o| > 3\sigma(|F_o|)$. The present study established the molecular structure of the title compound obtained from a temperature-dependent rearrangement reaction. The phenyl group at C(3) is not conjugated with the heterocyclic ring being almost perpendicular (92·3°) to it. There are no unusual intramolecular distances or angles.

Introduction. We have found that 5-methyl-11-[(4-methylphenyl)methylene-6,12-dioxodibenz[*b*,*f*]azocine (1) reacts with primary amines to give products from novel rearrangements, in good yield. For example aliphatic amines give diazachrysenes (Letcher, Ng & Cheung, 1987), and aniline at 403 K gives a dioxospiro[indole-2,1′-isoindole] derivative (Cheung, Letcher & Ng, 1989). The latter reaction is apparently temperature dependent since at 443 K, a different

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