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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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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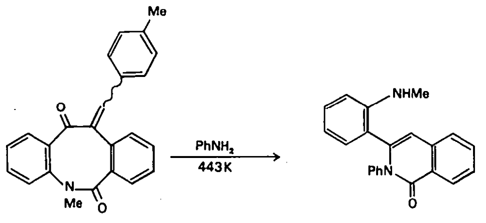
Table 1. *Atomic coordinates and isotropic thermal parameters* (Å²) *with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}
O	0·2532 (1)	0·1822 (1)	0·5002 (3)	5·46 (5)
N(1)	0·2711 (1)	0·3336 (1)	0·4586 (3)	4·01 (5)
N(2)	0·2344 (2)	0·5023 (2)	0·1375 (3)	7·26 (7)
C(1)	0·4243 (2)	0·1414 (2)	0·4559 (4)	5·61 (8)
C(2)	0·5074 (2)	0·1264 (2)	0·4310 (4)	6·88 (9)
C(3)	0·5597 (2)	0·2004 (2)	0·4081 (4)	7·14 (9)
C(4)	0·5286 (2)	0·2877 (2)	0·4087 (4)	6·48 (9)
C(5)	0·4436 (2)	0·3045 (2)	0·4307 (4)	4·75 (7)
C(6)	0·3916 (2)	0·2303 (2)	0·4547 (3)	4·27 (7)
C(7)	0·3020 (2)	2·2446 (2)	0·4755 (3)	4·18 (6)
C(8)	0·4075 (2)	0·3945 (2)	0·4279 (4)	5·17 (7)
C(9)	0·3246 (2)	0·4085 (2)	0·4415 (3)	4·35 (7)
C(10)	0·1775 (2)	0·3474 (2)	0·4460 (3)	4·19 (6)
C(11)	0·1478 (2)	0·3685 (2)	0·5897 (4)	5·86 (8)
C(12)	0·0574 (2)	0·3814 (2)	0·5707 (4)	7·42 (9)
C(13)	−0·0001 (2)	0·3722 (3)	0·4114 (5)	7·8 (1)
C(14)	0·0314 (2)	0·3497 (2)	0·2714 (5)	7·6 (1)
C(15)	0·1205 (2)	0·3370 (2)	0·2866 (4)	5·48 (8)
C(16)	0·2856 (2)	0·5011 (2)	0·4454 (4)	4·67 (7)
C(17)	0·2951 (2)	0·5456 (2)	0·6029 (4)	5·83 (8)
C(18)	0·2589 (2)	0·6317 (2)	0·6114 (4)	6·61 (8)
C(19)	0·2123 (2)	0·6716 (2)	0·4613 (4)	6·49 (8)
C(20)	0·2026 (2)	0·6296 (2)	0·3048 (4)	6·52 (8)
C(21)	0·2404 (2)	0·5429 (2)	0·2935 (4)	5·43 (7)
C(22)	0·1696 (3)	0·5293 (3)	−0·0150 (5)	12·0 (1)
H(N)	0·252 (2)	0·445 (2)	0·135 (3)	4·0 (7)

compound (2) is unexpectedly produced. The structure (2), deduced from spectroscopy together with several other possible structures, has now been confirmed in this X-ray crystallographic study. Details of the chemistry will be reported elsewhere.



(1) (2)

Experimental. The title compound (2) was obtained in 60% yield from the reaction between (1) and aniline at 443 K, and crystallized from chloroform–light petroleum (b.p. 40–60°).

Colourless crystal of dimensions 0·15 × 0·15 × 0·5 mm. Enraf–Nonius CAD-4 diffractometer, with graphite-monochromated Mo $K\alpha$ radiation, and 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 4794 reflections, $2\theta_{max} = 48°$, in the range $-17 \le h \le 17$, $-16 \le k \le 16$, $-9 \le l \le 9$, 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 0·4% 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, 2549 reflections were obtained, $R_{int} = 0{\cdot}023$; 1394 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, Fisk, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least squares (on F) with atomic scattering factors from *International Tables for X-ray Crystallography* (1974), using the *SDP* programs (Enraf–Nonius, 1985) on a MicroVAX II computer. The C, N and O atoms were refined anisotropically. The H(N) atom was located in the difference Fourier map and was refined isotropically. The other H atoms in calculated positions were not refined. Convergence for 230 variables by least squares with $w = 4F_o^2/[\sigma^2(F_o)^2 + (0{\cdot}04F_o^2)^2]$ was reached at $R = 0{\cdot}041$, $wR = 0{\cdot}045$ and $S = 1{\cdot}282$ for the 1394 reflections. $(\Delta/\sigma)_{max} = 0{\cdot}05$. A final difference Fourier map was featureless, with maximum positive and negative peaks of 0·13 and 0·15 e Å⁻³.

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

O–C(7)	1·235 (3)	C(8)–C(9)	1·337 (4)
N(1)–C(7)	1·384 (3)	C(9)–C(16)	1·490 (4)
N(1)–C(9)	1·405 (3)	C(10)–C(11)	1·370 (4)
N(1)–C(10)	1·452 (4)	C(10)–C(15)	1·373 (3)
N(2)–C(21)	1·360 (4)	C(11)–C(12)	1·395 (4)
N(2)–C(22)	1·437 (4)	C(12)–C(13)	1·374 (4)
C(1)–C(2)	1·373 (4)	C(13)–C(14)	1·362 (6)
C(1)–C(6)	1·398 (4)	C(14)–C(15)	1·378 (4)
C(2)–C(3)	1·393 (5)	C(16)–C(17)	1·390 (5)
C(3)–C(4)	1·368 (5)	C(16)–C(21)	1·387 (4)
C(4)–C(5)	1·400 (5)	C(17)–C(18)	1·389 (4)
C(5)–C(6)	1·395 (4)	C(18)–C(19)	1·372 (5)
C(5)–C(8)	1·430 (4)	C(19)–C(20)	1·365 (4)
C(6)–C(7)	1·459 (4)	C(20)–C(21)	1·411 (4)

C(7)–N(1)–C(9)	122·8 (3)	C(8)–C(9)–C(16)	123·3 (2)
C(7)–N(1)–C(10)	117·3 (3)	N(1)–C(10)–C(11)	120·6 (3)
C(9)–N(1)–C(10)	119·7 (2)	N(1)–C(10)–C(15)	117·8 (2)
C(21)–N(2)–C(22)	122·2 (3)	C(11)–C(10)–C(15)	121·7 (2)
C(2)–C(1)–C(6)	120·3 (3)	C(10)–C(11)–C(12)	118·4 (2)
C(1)–C(2)–C(3)	119·9 (4)	C(11)–C(12)–C(13)	120·5 (3)
C(2)–C(3)–C(4)	120·5 (3)	C(12)–C(13)–C(14)	119·8 (3)
C(3)–C(4)–C(5)	120·9 (3)	C(13)–C(14)–C(15)	121·0 (3)
C(4)–C(5)–C(6)	118·6 (3)	C(10)–C(15)–C(14)	118·8 (4)
C(4)–C(5)–C(8)	122·7 (3)	C(9)–C(16)–C(17)	119·3 (2)
C(6)–C(5)–C(8)	118·6 (2)	C(9)–C(16)–C(21)	120·5 (2)
C(1)–C(6)–C(5)	120·1 (2)	C(17)–C(16)–C(21)	120·2 (2)
C(1)–C(6)–C(7)	119·4 (2)	C(16)–C(17)–C(18)	120·8 (2)
C(5)–C(6)–C(7)	120·5 (2)	C(17)–C(18)–C(19)	118·7 (3)
O–C(7)–N(1)	119·8 (2)	C(18)–C(19)–C(20)	121·6 (3)
O–C(7)–C(6)	123·7 (2)	C(19)–C(20)–C(21)	120·5 (3)
N(1)–C(7)–C(6)	116·4 (2)	N(2)–C(21)–C(16)	121·0 (3)
C(5)–C(8)–C(9)	121·6 (3)	N(2)–C(21)–C(20)	120·9 (2)
N(1)–C(9)–C(8)	119·8 (2)	C(16)–C(21)–C(20)	118·2 (3)
N(1)–C(9)–C(16)	117·0 (2)		

C(9)–N(1)–C(7)–O	175·8 (2)	C(10)–N(1)–C(9)–C(16)	11·7 (3)
C(9)–N(1)–C(7)–C(6)	−6·9 (3)	C(22)–N(2)–C(21)–C(16)	162·3 (3)
C(10)–N(1)–C(7)–O	−8·7 (3)	C(22)–N(2)–C(21)–C(20)	−19·2 (5)
C(10)–N(1)–C(7)–C(6)	168·6 (2)	N(1)–C(9)–C(16)–C(17)	89·5 (3)
C(7)–N(1)–C(9)–C(8)	5·0 (4)	N(1)–C(9)–C(16)–C(21)	−90·7 (3)
C(7)–N(1)–C(9)–C(16)	−173·0 (2)	C(8)–C(9)–C(16)–C(17)	−88·4 (4)
C(10)–N(1)–C(9)–C(8)	−170·3 (2)	C(8)–C(9)–C(16)–C(21)	91·3 (4)

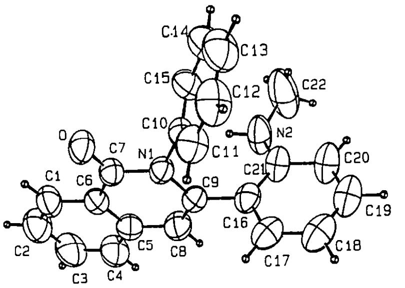


Fig. 1. A perspective view of the molecule (2).

Discussion. The *ORTEP* drawing (Johnson, 1976) of the molecule with atomic numbering scheme is shown in Fig. 1. The atomic parameters of the C, N, O atoms and the H(N) atom are listed in Table 1.* Bond lengths, bond angles and selected torsion angles are given in Table 2. The X-ray structural data reveal intermolecular hydrogen bonding between the NH and amide carbonyl groups, with NH, N···O, H···O distances and N—H···O angle of 0·89, 3·04, 2·15 Å and 150·3°, respectively.

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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 51515 (11 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 $[PhCH_2NMe_3]^+[B_{10}H_{13}]^-$

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Abstract. Benzyltrimethylammonium tridecahydro-*nido*-decaborate(1−), $[PhCH_2NMe_3]^+[B_{10}H_{13}]^-$, M_r = 271·5, monoclinic, $P2_1/c$, a = 8·8759 (13), b = 18·739 (3), c = 10·8215 (13) Å, β = 96·223 (11)°, V = 1789·3 Å^3, Z = 4, D_x = 1·008 Mg m^{-3}, Mo $K\alpha$, λ = 0·71069 Å, μ = 0·046 mm^{-1}, $F(000)$ = 584, room temperature, R = 0·0524, based on 1581 unique observed reflections. The structural effects of deprotonation are shortenings of the B(9)—B(10) and B(5)—B(10) connectivities, and these changes are rationalized by the results of a molecular orbital calculation.

Introduction. We are currently investigating (Wynd, McLennan, Reed & Welch, 1987; Wynd, Parish & Welch, 1989) how the introduction of $\{AuPR_3\}$ (R = alkyl) fragments into $\{B_{10}\}$ cages affects the electronic and consequent geometric structure of the cage. The analysis of the metallaborane cage architecture relies mainly on comparison with the structures of analogous boranes. Whilst the literature contains a communication (Sneddon, Huffman, Schaeffer & Streib, 1972) reporting the structure of the $[B_{10}H_{13}]^-$ anion, as its $[NHEt_3]^+$ salt, no fractional coordinates were published or deposited, nor are any available from the authors. The structure of the isoelectronic, isovertex species, $B_{10}H_{14}$, is known to a high degree of accuracy (Brill, Dietrich & Dierks, 1971). However since the effects of deprotonation can substantially modify the internuclear distances in such species (Mitchell & Welch, 1987), we have determined the structure of $[PhCh_2NMe_3]^+$ $[B_{10}H_{13}]^-$ (1). This enables useful comparisons to be drawn against the structures of other boranes and metallaborane derivatives (this paper; Wynd, Parish & Welch, 1989). (1) was synthesized using a method based on literature preparations (Hawthorne, Pitochelli, Strahm & Miller, 1960).

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