$V = 1389\ (1)$ Å^3
$Z = 4$
$D_x = 1.24$ Mg m^{-3}

Needle
$0.46 \times 0.32 \times 0.14$ mm
Colourless

Data collection

Nicolet P3/F diffractometer
$2\theta/\theta$ scans
Absorption correction: none
1052 measured reflections
930 independent reflections
878 observed reflections
$[I > 2.5\sigma(I)]$

$\theta_{max} = 55°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 12$
2 standard reflections
monitored every 50
reflections
intensity variation: 3%

Refinement

Refinement on F
$R = 0.034$
$wR = 0.046$
$S = 1.30$
878 reflections
175 parameters
$w = [\sigma^2(F_o) + 0.07(F_o)^2]^{-1}$

$(\Delta/\sigma)_{max} = 0.70$
$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.11$ e Å^{-3}
Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV)

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

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O(1)	0.0425 (2)	0.3937 (2)	0.3419	4.8 (1)
O(2)	0.0870 (3)	0.0234 (2)	−0.1977 (4)	7.2 (2)
O(3)	−0.2376 (3)	0.1382 (1)	−0.1192 (3)	5.3 (1)
C(1)	−0.0583 (3)	0.1515 (2)	0.0375 (4)	4.0 (1)
C(2)	−0.0004 (3)	0.2482 (2)	0.0765 (4)	3.3 (1)
C(3)	−0.0134 (3)	0.2761 (2)	0.1934 (4)	3.6 (1)
C(4)	0.0505 (3)	0.3610 (2)	0.2294 (3)	3.6 (1)
C(5)	0.1301 (3)	0.4202 (2)	0.1529 (3)	3.6 (1)
C(6)	0.1387 (3)	0.3918 (2)	0.0379 (3)	3.6 (1)
C(7)	0.0744 (3)	0.3077 (2)	−0.0029 (3)	3.4 (1)
C(8)	0.0818 (3)	0.2844 (2)	−0.1304 (4)	4.1 (1)
C(9)	0.0228 (3)	0.1857 (2)	−0.1692 (4)	3.9 (1)
C(10)	0.1517 (4)	0.1096 (2)	−0.1551 (4)	4.9 (2)
C(11)	0.2048 (3)	0.0992 (2)	−0.0304 (4)	4.9 (2)
C(12)	0.0703 (4)	0.0766 (2)	0.0498 (4)	4.8 (2)
C(13)	−0.1057 (3)	0.1567 (2)	−0.0869 (4)	3.8 (1)
C(14)	0.2045 (4)	0.5104 (2)	0.1950 (4)	5.3 (2)
C(15)	−0.0295 (5)	0.1907 (3)	−0.2951 (4)	5.5 (2)
C(16)	−0.0430 (5)	0.3396 (3)	0.4234 (4)	6.2 (2)

Table 2. *Bond lengths* (Å) *and angles* (°)

O(1)—C(4)	1.376 (4)	C(5)—C(6)	1.384 (5)
O(1)—C(16)	1.413 (5)	C(5)—C(14)	1.499 (4)
O(2)—C(10)	1.419 (5)	C(6)—C(7)	1.387 (4)
O(3)—C(13)	1.221 (3)	C(7)—C(8)	1.505 (4)
C(1)—C(2)	1.514 (4)	C(8)—C(9)	1.542 (5)
C(1)—C(12)	1.532 (5)	C(9)—C(10)	1.548 (4)
C(1)—C(13)	1.490 (5)	C(9)—C(13)	1.512 (5)
C(2)—C(3)	1.406 (5)	C(9)—C(15)	1.519 (6)
C(2)—C(7)	1.395 (4)	C(10)—C(11)	1.513 (6)
C(3)—C(4)	1.376 (4)	C(11)—C(12)	1.513 (6)
C(4)—C(5)	1.392 (4)		
C(4)—O(1)—C(16)	118.1 (3)	C(6)—C(7)—C(8)	119.9 (3)
C(2)—C(1)—C(12)	110.6 (2)	C(7)—C(8)—C(9)	117.6 (3)
C(2)—C(1)—C(13)	109.3 (3)	C(8)—C(9)—C(10)	110.8 (2)
C(12)—C(1)—C(13)	108.6 (3)	C(8)—C(9)—C(13)	107.5 (3)
C(1)—C(2)—C(3)	120.6 (3)	C(8)—C(9)—C(15)	109.4 (3)
C(1)—C(2)—C(7)	119.6 (3)	C(10)—C(9)—C(13)	105.7 (3)
C(3)—C(2)—C(7)	119.7 (3)	C(10)—C(9)—C(15)	110.1 (3)
C(2)—C(3)—C(4)	119.9 (3)	C(13)—C(9)—C(15)	113.2 (3)
O(1)—C(4)—C(3)	123.6 (3)	O(2)—C(10)—C(9)	105.9 (2)

O(1)—C(4)—C(5)	114.9 (2)	O(2)—C(10)—C(11)	111.3 (3)
C(3)—C(4)—C(5)	121.5 (3)	C(9)—C(10)—C(11)	112.5 (3)
C(4)—C(5)—C(6)	117.3 (2)	C(10)—C(11)—C(12)	111.6 (2)
C(4)—C(5)—C(14)	120.7 (3)	C(1)—C(12)—C(11)	110.5 (3)
C(6)—C(5)—C(14)	122.1 (3)	O(3)—C(13)—C(1)	122.5 (3)
C(5)—C(6)—C(7)	123.3 (3)	O(3)—C(13)—C(9)	123.0 (3)
C(2)—C(7)—C(6)	118.1 (3)	C(1)—C(13)—C(9)	114.5 (2)
C(2)—C(7)—C(8)	121.9 (3)		

Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods (*TEXSAN*; Molecular Structure Corporation, 1990). The H-atom positions in the CH, CH$_2$ and CH$_3$ groups were allowed to ride on the bonded C atoms and refined. The H atom bonded to the O atom was located on a difference Fourier map at an advanced stage of anisotropic refinement and its coordinates refined.

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Structure of 2,4,6-Tri(*tert*-butyl)aniline at 153 K

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Abstract

The structure of 2,4,6-tri(*tert*-butyl)aniline is reported. The molecule lies on a crystallographic twofold axis.

Comment

Alkoxides and amides of the later transition metals are often difficult to study as they tend towards extensive association and low solubility in hydrocarbon solvents (Bradley, Mehrota & Gaur, 1978). The use of sterically demanding substituents, such as adamantyl, triphenyl-methyl or 2,4,6-tri(*tert*-butyl)phenyl, reduces alkoxide and amide bridging and improves solubility (Bartlett, Ellison, Power & Shoner, 1991). 2,4,6-Tri(*tert*-butyl)-aniline thus could be a suitable ligand to reduce association in metal amides. The molecule lies on a crystallographic twofold axis. The disordered *para-tert*-butyl group was refined with distance restraints for the 1-2 and 1-3 distances to an occupancy of 0.5:0.5 for the two positions. The position of the amino H atom was refined with a distance restraint for the N—H bond length. All bond lengths and angles are generally as expected.
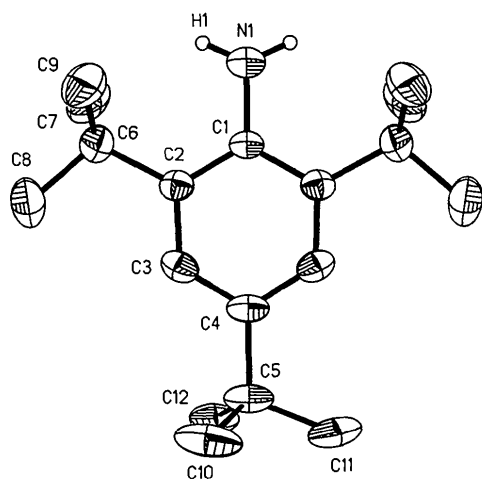

Fig. 1. Structure of the title compound showing 50% probability displacement ellipsoids. The H atoms except H1 are omitted for clarity.

Experimental

Crystal data

$C_{18}H_{31}N$
$M_r = 261.44$
Orthorhombic
Pnma
$a = 9.2210\ (10)$ Å
$b = 18.157\ (2)$ Å
$c = 10.3180\ (10)$ Å
$V = 1727.5\ (3)$ Å^3
$Z = 4$
$D_x = 1.005$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 64 reflections
$\theta = 10-12.5°$
$\mu = 0.057$ mm^{-1}
$T = 153\ (2)$ K
Prism
$0.4 \times 0.3 \times 0.3$ mm
Colorless

Data collection

Stoe Siemens AED four-circle diffractometer
$R_{int} = 0.0246$
$\theta_{max} = 24.98°$

Profile data from $2\theta/\omega$ scans
Absorption correction: none
2149 measured reflections
1567 independent reflections
1321 observed reflections
$[I > 2\sigma(I)]$

$h = -3 \rightarrow 10$
$k = -21 \rightarrow 21$
$l = -12 \rightarrow 12$
3 standard reflections
frequency: 90 min
intensity variation: none

Refinement

Refinement on F^2
$R[F>4\sigma(F)] = 0.052$
$wR(F^2) = 0.166$
$S = 1.086$
1564 reflections
116 parameters
Only coordinates of H atoms refined

Calculated weights
$w = 1/[\sigma^2(F_o^2)+(0.0799P)^2 +0.5768P]$
where $P = (F_o^2 + 2F_c^2)/3$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Refinement on F^2 for all reflections except for three with very negative F^2 or flagged by the user for potential systematic errors. Data collection: *DIF*4 (Stoe & Cie, 1988a). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988b). Program(s) used to solve structure: *SHELXS*-90 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*-92 (Sheldrick, 1992). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL*-92.

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

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

	x	y	z	U_{eq}
N1	0.0978 (2)	0.7500	0.3241 (2)	0.0423 (10)
C1	0.2317 (2)	0.7500	0.3911 (2)	0.0282 (10)
C2	0.2962 (2)	0.68253 (8)	0.42781 (13)	0.0314 (7)
C3	0.4273 (2)	0.68500 (8)	0.49517 (14)	0.0346 (8)
C4	0.4961 (2)	0.7500	0.5294 (2)	0.0336 (10)
C6	0.2234 (2)	0.60754 (9)	0.4007 (2)	0.0478 (10)
C7	0.2100 (3)	0.59136 (11)	0.2551 (2)	0.0711 (13)
C8	0.3113 (3)	0.54316 (11)	0.4590 (3)	0.0870 (15)
C9	0.0732 (2)	0.60425 (11)	0.4673 (2)	0.0647 (11)
C5	0.6405 (2)	0.7500	0.6046 (2)	0.0433 (10)
C10	0.6210 (4)	0.7132 (3)	0.7336 (3)	0.070 (2)
C11	0.6941 (4)	0.8324 (2)	0.6332 (4)	0.057 (2)
C12	0.7568 (3)	0.7152 (2)	0.5246 (4)	0.053 (2)

Table 2. *Geometric parameters* (Å, °)

N1—C1	1.415 (3)	C6—C7	1.536 (3)
N1—H1	0.921 (9)	C6—C8	1.544 (3)
C1—C2	1.413 (2)	C6—C9	1.547 (3)
C2—C3	1.396 (2)	C5—C12	1.494 (4)
C2—C6	1.544 (2)	C5—C10	1.500 (4)
C3—C4	1.386 (2)	C5—C11	1.603 (4)
C4—C5	1.541 (3)		
C1—N1—H1	114 (2)	C2—C6—C8	111.7 (2)
C2^i—C1—C2	120.2 (2)	C7—C6—C9	110.8 (2)
C2—C1—N1	119.9 (1)	C2—C6—C9	110.1 (2)
C3—C2—C1	118.0 (1)	C8—C6—C9	105.6 (2)
C3—C2—C6	119.7 (1)	C12—C5—C10	112.9 (2)
C1—C2—C6	122.2 (2)	C12—C5—C4	110.0 (2)
C4—C3—C2	123.4 (2)	C10—C5—C4	110.1 (2)
C3—C4—C5	121.6 (1)	C12—C5—C11	105.9 (2)
C7—C6—C2	112.4 (2)	C10—C5—C11	106.8 (3)
C7—C6—C8	106.1 (2)	C4—C5—C11	111.1 (2)

Symmetry code: (i) $x, \tfrac{3}{2} - y, z$.

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Etude de la Phenylhydrazone du 3-Benzoyl-1-methylindole

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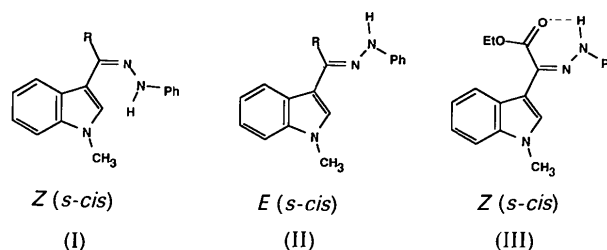
Abstract

The Z(*s-cis*) isomer of 3-benzoyl-1-methylindole phenylhydrazone derived from a 3-aroyl-1-methylindole, was isolated. The structure consists of two molecules with similar conformations in the asym-

metric unit. The dihedral angle ($\simeq 55°$) between the methyl indole substituent and the hydrazone moiety leads to a break in the delocalization.

Commentaire

Nous avons proposé dans un travail antérieur (Soufiaoui, Laude & Arriau, 1977) que les arylhydrazones des 3-aroyl-1-méthylindoles existent en solution, d'après les observations spectroscopiques, sous deux configurations E et Z difficilement séparables par chromatographie ou par recristallisation fractionnée. Ayant isolé un monocristal d'une seule forme ($R = Ph$), nous avons soumis ce dernier à une étude en diffraction X. Cette forme correspond à la configuration Z (R en *trans* de NHPh) (I) prédominante en solution. En substituant R par un groupement éthoxycarbonyl, c'est la forme Z (CO_2Et en *cis* de NHPh) (III) qui prédomine en raison de la liaison hydrogène (Ruccia, Vivona, Piozzi & Aversa, 1969) et qui correspond à E pour $R = Ph$.

Z (*s-cis*)   E (*s-cis*)   Z (*s-cis*)
  (I)   (II)   (III)

La Fig. 1 montre une vue *ORTEP* (Johnson, 1965) numérotée des deux molécules qui constituent le motif élémentaire. Au vu de ces résultats on peut remarquer une bonne homogénéité dans la géométrie globale des deux molécules constituant le motif. Cependant ces molécules ne sont pas planes à l'état solide: comme le montre la Fig. 2, le fragment phénylhydrazone de chaque molécule est plan mais forme un angle important avec la partie méthylindole [respectivement 124,5 (3) et 125,3 (3)°] du fait d'effets stériques déterminants dans la rupture de conjugaison. Ces molécules diffèrent légèrement par leur
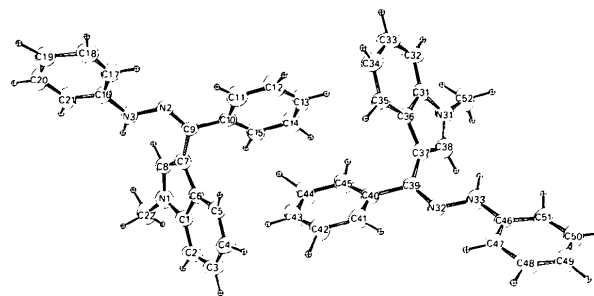

Fig. 1. Vue *ORTEP* (Johnson, 1965) du composé (2).