asymmetric in that the Se—C(1)—C(2) and Se—C(1)—C(6) bond angles are 123·7 (2) and 115·6 (2)°, respectively, and the Se—C bond is out of the phenyl plane. Studies of phenoxyalkanoic acid derivatives (Kennard, Smith & White, 1982) and hexakis-(methylthio)benzene, $C_6(SCH_3)_6$ (Peach & Burschka, 1982), showed similar behavior in O— and S–phenyl bonds. For O and S the deviation from the expected trigonal arrangement was thought to be due to crowding, but this work leads us to conclude that very weak forces can cause this type of distortion, hence the change is not chemically significant.

Analysis of the thermal-motion parameters (THMV9, Trueblood, 1985; Rosenfield, Trueblood & Dunitz, 1978) was carried out in an attempt to identify librating groups of atoms. Neither the nitro group nor the phenyl ring was indicated to be librating separately from the rest of the molecule. The relative motions of atoms in different groups of the molecule were not significantly greater than those within a group, hence separately librating groups were not suggested by the analysis. Interatomic distances corrected for thermal motion were calculated treating the whole molecule as a rigid body and are listed in Table 2. The corrections were all less than σ.

A weak intermolecular H bond may exist between H(6) and O(1). The H—O distance is 2·45 (4) Å, the O(1)—H(6)—C(6) angle 157 (3)° and the N—O(1)—H(6) angle 158 (1)°. The interacting molecules are related by the C centering.

References

BENT, H. A. (1961). *J. Chem. Phys.* **33**, 1259–1260.
DAVIES, E. K. (1984). *SNOOPI* plot program. Chemical Crystallography Laboratory, Oxford Univ.
GABE, E. J., LARSON, A. C., LEE, F. L. & LE PAGE, Y. (1984). *NRC VAX Crystal Structure System.* National Research Council of Canada, Ottawa.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
KENNARD, C. H. L., SMITH, G. & WHITE, A. H. (1982). *Acta Cryst.* **B38**, 868–875.
PEACH, M. E. & BURSCHKA, C. (1982). *Can. J. Chem.* **60**, 2029–2037.
ROSENFIELD, R. E. JR, TRUEBLOOD, K. N. & DUNITZ, J. D. (1978). *Acta Cryst.* **A34**, 828–829.
TRUEBLOOD, K. N. (1985). *THMV9* thermal-motion-analysis program. Private communication.
WOODARD, C. M., BROWN, D. S., LEE, J. D. & MASSEY, A. G. (1976). *J. Organomet. Chem.* **121**, 333–344.

Structure of 3-Benzyl-2-methylindole

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Abstract. $C_{16}H_{15}N$, $M_r = 221·3$, monoclinic, $P2_1/n$, $a = 12·458$ (1), $b = 5·904$ (1), $c = 17·293$ (2) Å, $\beta = 102·75$ (1)°, $V = 1240·6$ (3) Å^3, $Z = 4$, $D_m = 1·180$, $D_x = 1·184$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0·7107$ Å, $\mu = 0·56$ cm^{-1}, $F(000) = 472$, $T = 296$ K, $R = 0·054$ for 1084 observed reflections. The indole system is nearly planar. The methyl group is slightly out of the plane of the indole system. The substituent benzene ring makes a dihedral angle of 79·8 (1)° with the pyrrole ring.

Introduction. Indole and its various substituted products have long been known for their interesting chemical and biological activities. Our interest in the

structural properties of 3-benzyl-2-methylindole is twofold: (1) its structural study might aid in the elucidation of its mechanistic action; (2) there is, somewhat surprisingly, relatively little knowledge about the crystal chemistry of non-steroidal indole-based derivatives. As an aid in understanding the above mentioned mechanistic studies and to provide further structural data on substituted indoles, we have investigated the solid-state structure of the title compound by X-ray diffraction methods and give a full report of the results here.

Experimental. Single crystals of the title compound obtained by slow evaporation of the powder substance in chloroform. The approximate dimensions of the

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selected crystal were $0.2 \times 0.3 \times 0.18$ mm. Intensities measured by $\omega-2\theta$ scan method, $0° \le 2\theta \le 45°$, Picker diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. Density by flotation using aqueous KI solution. Unit-cell constants derived by least-squares analysis of 25 reflections for which $25° < 2\theta < 38°$. Range of h,k,l: $h=0\rightarrow13$, $k=0\rightarrow6$, $l=-18\rightarrow18$. Out of 1630 unique reflections 546 had intensities less than $3\sigma(I)$. They were classed as unobserved and excluded from subsequent refinement. Lorentz and polarization corrections were applied but no absorption correction.

H atoms associated with the benzene ring were generated from the known geometry around C atoms; other H atoms located from the difference map and given same isotropic temperature factors as the atoms to which they were bonded. The structure was solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Refinement carried out first with isotropic temperature factors and then with anisotropic temperature factors for non-hydrogen atoms until $R = 0.054$ and $wR = 0.068$ with 154 parameters. $S = 2.25$. Function minimized during refinement was $\sum w(\Delta F)^2$, $w = 1/\sigma^2(F_o)$. H atoms not refined and not included in the final step of refinement. Max. least-squares shift-to-error ratio is 0.074. Average shift-to-error ratio is 0.12; max. and min. heights in final difference map ± 0.6 e Å^{-3}. Scattering factors for non-hydrogen atoms from *International Tables for X-ray Crystallography* (1974) and for H atoms from Stewart, Davidson & Simpson (1965). No corrections for secondary extinction. Computations carried out at Univ. of California using *UCLA Crystallographic Computing Package* and also on the Burroughs B6700 computer system, Calcutta, with modified programs from *XRAY ARC* (Vickery, Bright & Mallinson, 1973).

Discussion. The atomic coordinates, bond lengths and bond angles with their estimated standard deviations are given in Tables 1 and 2.* From the analysis of planarity of the indole system [which includes the C(4)–C(9) benzene ring fused with the N(1), C(2)–C(4), C(9) pyrrole ring] it has been found that the whole system is nearly planar $[\sum(\Delta/\sigma)^2 = 47.8]$ and that the largest displacements of the atoms from the plane are -0.013 (4) Å for C(6), 0.011 (4) Å for C(3) and -0.010 (4) Å for C(2). C(10) is -0.083 (5) Å out of the indole plane.

The dihedral angle between the pyrrole ring and the substituent benzene ring is 79.8 (1)°. The planarity of

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43046 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
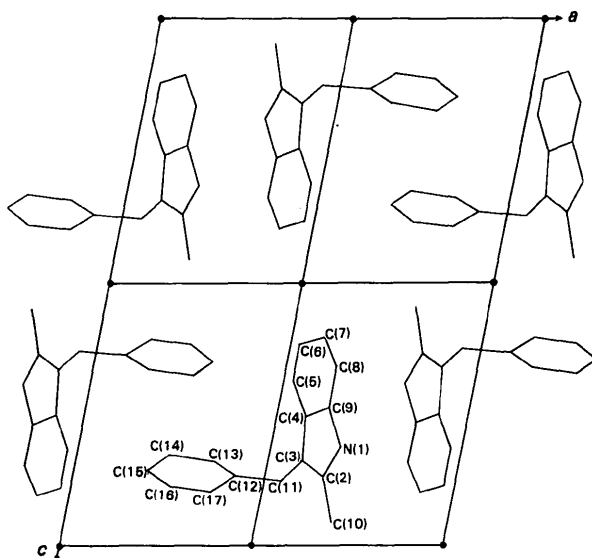
the C(9), N(1), C(2) and C(10) system and shortening of N(1)–C(2) = 1.388 (5) Å are due to the delocalized π-electron system of the benzenoid ring which extends over the atoms N(1), C(2) and C(10). A packing diagram is shown in Fig. 1. Layers of molecules are displaced with respect to one another to relieve short intermolecular contacts. The structure consists of individual molecules packed in the crystal *via* van der Waals forces. There are no unusual short intermolecular contacts, and hence the packing forces are unlikely to influence the geometry of the molecule to an appreciable extent.

Table 1. *Fractional atomic coordinates with e.s.d.'s and isotropic thermal parameters* (Å^2)

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

	x	y	z	B_{eq}
C(2)	0.6541 (3)	0.1801 (6)	0.8691 (2)	4.6
C(3)	0.5914 (3)	0.3619 (6)	0.8379 (2)	4.0
C(4)	0.5778 (2)	0.3475 (6)	0.7532 (6)	3.9
C(5)	0.5248 (3)	0.4840 (6)	0.6898 (2)	4.9
C(6)	0.5286 (3)	0.4184 (7)	0.6134 (2)	5.6
C(7)	0.5824 (3)	0.2213 (8)	0.5988 (2)	5.7
C(8)	0.6359 (3)	0.0850 (7)	0.6602 (2)	5.2
C(9)	0.6328 (3)	0.1504 (6)	0.7371 (2)	4.2
C(10)	0.6986 (4)	0.1151 (7)	0.9542 (2)	6.7
C(11)	0.5471 (3)	0.5465 (6)	0.8815 (2)	4.8
C(12)	0.4222 (3)	0.5417 (6)	0.8705 (2)	4.2
C(13)	0.3592 (3)	0.7251 (6)	0.8362 (2)	5.3
C(14)	0.2453 (4)	0.7211 (7)	0.8265 (2)	6.0
C(15)	0.1939 (3)	0.5383 (8)	0.8510 (2)	5.7
C(16)	0.2556 (3)	0.3545 (7)	0.8853 (2)	5.4
C(17)	0.3699 (3)	0.3556 (6)	0.8947 (2)	4.6
N(1)	0.6799 (2)	0.0485 (5)	0.8092 (2)	4.9

Table 2. *Bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

C(2)–N(1)	1.388 (5)	C(2)–C(10)	1.503 (5)
C(2)–C(3)	1.366 (5)	C(3)–C(11)	1.498 (5)
C(3)–C(4)	1.439 (5)	C(11)–C(12)	1.526 (5)
C(4)–C(5)	1.403 (5)	C(12)–C(13)	1.391 (5)
C(5)–C(6)	1.388 (5)	C(13)–C(14)	1.392 (6)
C(6)–C(7)	1.393 (6)	C(14)–C(15)	1.369 (6)
C(7)–C(8)	1.381 (5)	C(15)–C(16)	1.385 (6)
C(8)–C(9)	1.393 (5)	C(16)–C(17)	1.397 (5)
C(4)–C(9)	1.409 (5)	C(12)–C(17)	1.388 (5)
N(1)–C(9)	1.392 (5)		

N(1)–C(2)–C(3)	110.6 (3)	N(1)–C(9)–C(4)	107.9 (3)
N(1)–C(2)–C(10)	119.4 (3)	C(2)–N(1)–C(9)	107.8 (3)
C(3)–C(2)–C(10)	130.0 (3)	C(3)–C(11)–C(12)	113.7 (3)
C(4)–C(3)–C(11)	125.5 (3)	C(11)–C(12)–C(13)	120.2 (3)
C(2)–C(3)–C(4)	106.5 (3)	C(11)–C(12)–C(17)	120.7 (3)
C(2)–C(3)–C(11)	127.9 (3)	C(13)–C(12)–C(17)	119.1 (3)
C(3)–C(4)–C(5)	133.7 (3)	C(12)–C(13)–C(14)	120.2 (3)
C(3)–C(4)–C(9)	107.3 (3)	C(13)–C(14)–C(15)	120.7 (4)
C(5)–C(4)–C(9)	119.0 (3)	C(14)–C(15)–C(16)	119.8 (4)
C(4)–C(5)–C(6)	118.3 (3)	C(15)–C(16)–C(17)	120.1 (3)
C(5)–C(6)–C(7)	121.6 (4)	C(12)–C(17)–C(16)	120.2 (3)
C(6)–C(7)–C(8)	121.1 (4)		
C(7)–C(8)–C(9)	117.5 (3)		
C(4)–C(9)–C(8)	122.3 (3)		

Fig. 1. Molecular projection of the title compound viewed down [010].

References

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht).
MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
VICKERY, B. L., BRIGHT, D. & MALLINSON, P. R. (1973). *XRAY ARC* program system. Agricultural Research Council, London.

Structure and Stable Conformations of a New Analgesic: 4-[5-Ethyl-4-(4-methoxyphenyl)-4H-1,2,4-triazol-3-yl]-N,N-dimethylbenzamine

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Abstract. $C_{19}H_{22}N_4O$, a powerful analgesic compound, $M_r = 322.4$, monoclinic, $P2_1/c$, $a = 18.237$ (3), $b = 8.494$ (3), $c = 12.340$ (2) Å, $\beta = 108.01$ (2)°, $V = 1817.9$ (8) Å^3, $Z = 4$, $D_x = 1.178$ g cm^{-3}, $\lambda(CuK\bar{\alpha}) = 1.5418$ Å, $\mu = 9.7$ cm^{-1}, $F(000) = 688$, $T = 298$ K, final $R = 0.072$ for 1878 independent observed reflections. Crystal cohesion is based on numerous van der Waals interactions. The mean planes of the 4-methoxyphenyl and 4-dimethylaminophenyl groups make angles of 79 and 28°, respectively, with the triazole ring. Conformational analysis of the isolated molecule shows on the one hand that the most stable conformation is somewhat different from the crystalline structure and on the other hand that both phenyl rings have a large degree of conformational freedom.

Introduction. Pharmacological studies of numerous 1,2,4-triazole derivatives have shown that some of them are endowed with interesting analgesic properties (Clémence *et al.*, 1985) whilst being devoid of anti-inflammatory activity that would reduce their tolerance at the gastrointestinal level. Numerous thorough pharmacological and physicochemical studies have been performed on the most promising compound (RU 39813). We present here the crystal structure of the title compound and conformational analysis of the isolated molecule using semi-empirical calculations (CNDO/2).

Experimental. Title compound synthesized and crystallized by Roussel–Uclaf Laboratories (Romainville). Colourless, prismatic crystals, $0.6 \times 0.3 \times 0.2$ mm. Enraf–Nonius CAD-4 diffractometer with graphite monochromator; cell dimensions refined by least-squares fitting of θ values of 25 reflections ($15 \leq 2\theta \leq 42°$); intensities of 3090 unique reflections; $h \leq 21$, $k \leq 10$, $l \leq 13$; $\omega/2\theta$ scans ($2\theta < 120°$); $\theta = (0.8 + 0.15 \tan\theta)°$, detector aperture $(1.3 + 0.6 \tan\theta)$ mm; two reflections (321 and 610) monitored periodically