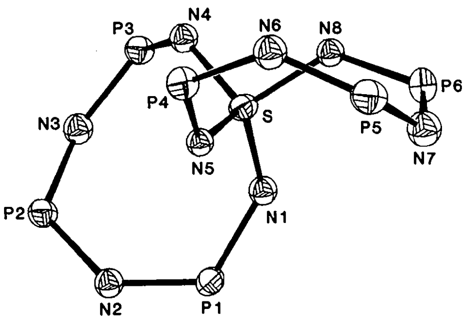

Fig. 1. *ORTEP*II diagram (Johnson, 1976) with 30% ellipsoids for the ring atoms (phenyl rings omitted for figure clarity).

Related literature. The spirocyclic nature of the structure is to be compared with that of $(Ph_4P_2N_3)_2S$ (Chivers, Rao & Richardson, 1985) and $(Ph_6P_3N_4)$-$S(S_2N_3)$ (Cordes, Oakley & Morito, 1988). The tub and saddle conformations of the two eight-membered rings are in keeping with the conformational motifs of sterically crowded cyclophosphazenes (Paddock, 1964; Bullen & Tucker, 1972; Bullen & Dann, 1974).

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References

Bullen, G. J. & Dann, P. E. (1974). *J. Chem. Soc. Dalton Trans.* pp. 705–709.

Bullen, G. J. & Tucker, P. A. (1972). *J. Chem. Soc. Dalton Trans.* pp. 1651–1658, 2437–2442.

Chivers, T., Rao, M. N. S. & Richardson, J. F. (1985). *Inorg. Chem.* **24**, 2237–2243.

Cordes, A. W., Oakley, R. T. & Morito, E. (1988). *Acta Cryst.* C**44**, 1838–1840.

Enraf–Nonius (1982). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Paddock, N. L. (1964). *Q. Rev. Chem. Soc.* **18**, 168–210.

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Structure of 2-*tert*-Butyl-4-methyl-2,4-dihydropyrrolo[3,4-*b*]indole

By Hans Preut, Gerald Dyker and Richard P. Kreher

Fachbereich Chemie, Universität Dortmund, D-4600 Dortmund 50, Postfach 500500, Federal Republic of Germany

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Abstract. $C_{15}H_{18}N_2$, $M_r = 226{\cdot}32$, orthorhombic, *Pbca*, $a = 9{\cdot}278$ (5), $b = 16{\cdot}334$ (7), $c = 17{\cdot}498$ (11) Å, $V = 2652$ (2) Å^3, $Z = 8$, $D_x = 1{\cdot}134$ Mg m^{-3}, $F(000) = 976$, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}06$ mm^{-1}, $T = 291$ (1) K, final $R = 0{\cdot}073$ for 1095 unique observed $[F \geq 3{\cdot}0\sigma(F)]$ diffractometer data. The three rings of the fused hetarene are nearly planar. The position of the methyl C atom at the N atom of the pyrrole ring does not deviate significantly from the least-squares plane through this central pyrrole ring. Thus the N atom may be considered sp^2-hybridized. According to the results of this analysis the molecule is to be regarded as a conjugated heteroaromatic system with 14 π electrons. There are no interactions between the molecules exceeding van der Waals forces.

Experimental. The tricyclic hetarene was prepared by the rational and efficient method of Kreher & Dyker (1987). In order to characterize the molecular geometry of the heterocyclic $(4n + 2)\pi$ system and to compare the experimental parameters with calculated values (CNDO) the crystal structure was determined. The planarity of the tricyclic hetarene is an essential feature for delocalization; distortion should be favourable for localized five- and six-membered π systems. The compound was crystallized from n-pentane at 248 K. Crystal size $0{\cdot}47 \times 0{\cdot}26 \times 0{\cdot}32$ mm, $\omega/2\theta$ scan, scan speed $1{\cdot}54$–$3{\cdot}33°$ min^{-1} in θ, Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$; lattice parameters from least-squares fit with 25 reflections up to $2\theta = 24{\cdot}5°$; six standard reflections recorded every 2·5 h, only random deviations; 5190 reflections measured, $1{\cdot}5 \leq \theta \leq 25{\cdot}0°$, $-11 \leq h \leq 11$, $0 \leq k \leq 19$, $0 \leq l \leq 20$; after averaging ($R_{\text{int}} = 0{\cdot}043$) 2329 unique reflections, 1095 with $F \geq 3{\cdot}0\sigma(F)$; Lorentz–polarization correction, no absorption correction; systematic absences $(0kl)\ k = 2n + 1$, $(h0l)\ l = 2n + 1$, $(hk0)\ h = 2n + 1$ conform to space group *Pbca;* structure solution *via* direct methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C—H 0·96 Å); refinement on F with 1095 reflections and 155 refined parameters; $w = 1{\cdot}0/[\sigma^2(F) + (0{\cdot}0005F^2)]$; $S = 1{\cdot}67$, $R = 0{\cdot}073$, $wR = 0{\cdot}065$, $(\Delta/\sigma)_{\max} = 0{\cdot}07$, no extinction correction; largest peak in final ΔF map $\pm 0{\cdot}2\,(1)\,\mathrm{e\,\AA^{-3}}$, atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974). A possible cause for the high R value could be the fact that the crystal diffracted weakly. Only 47% of the unique reflections have $F \geq 3{\cdot}0\sigma(F)$. Programs: Enraf–Nonius *Structure Determination Package* (Frenz, 1985), *PARST* (Nardelli, 1983), *SHELXTL PLUS* (Sheldrick, 1987), *SCHAKAL* (Keller, 1986). The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit cell in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1.* Bond lengths and bond angles, least-squares planes and torsion angles are given in Table 2. The three rings of the fused hetarene are nearly planar. The position of the methyl C atom at the N atom of the pyrrole ring does not deviate significantly from the least-squares plane through this central pyrrole ring. Thus the N atom may be considered sp^2-hybridized. According to the results of this analysis the molecule is to be regarded as a conjugated heteroaromatic system with 14 π electrons. CNDO calculations (Heitkamp, 1988), however, suggest a higher sp^3 character for the

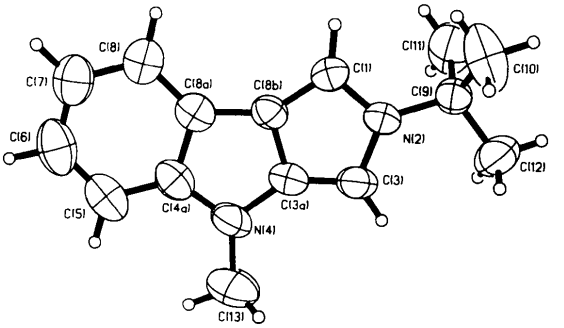

Fig. 1. General view (*SHELXTL PLUS* graphic) of the molecule, showing the atom-numbering scheme.

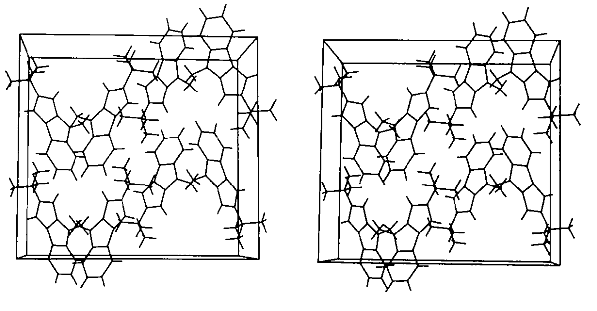

Fig. 2. Stereoscopic view (*SCHAKAL*) of the unit cell (**b** vertical, **c** horizontal).

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^3)

$$U_{\mathrm{eq}} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}
C(1)	0·0014 (5)	0·6899 (3)	0·4550 (3)	55
N(2)	0·0624 (4)	0·7640 (2)	0·4373 (2)	57
C(3)	0·1509 (5)	0·7574 (3)	0·3740 (3)	63
C(3a)	0·1460 (5)	0·6768 (3)	0·3543 (3)	55
N(4)	0·2029 (4)	0·6241 (3)	0·2994 (2)	70
C(4a)	0·1432 (5)	0·5474 (3)	0·3120 (3)	63
C(5)	0·1601 (6)	0·4774 (4)	0·2682 (3)	80
C(6)	0·0839 (7)	0·4091 (4)	0·2899 (3)	84
C(7)	−0·0066 (6)	0·4094 (3)	0·3523 (3)	75
C(8)	−0·0234 (5)	0·4798 (3)	0·3965 (3)	65
C(8a)	0·0504 (5)	0·5505 (3)	0·3761 (3)	53
C(8b)	0·0535 (4)	0·6334 (3)	0·4034 (2)	48
C(9)	0·0228 (5)	0·8425 (3)	0·4748 (3)	61
C(10)	0·0427 (7)	0·8337 (3)	0·5600 (3)	121
C(11)	−0·1342 (6)	0·8599 (3)	0·4580 (4)	106
C(12)	0·1173 (6)	0·9107 (3)	0·4477 (4)	138
C(13)	0·3027 (6)	0·6482 (3)	0·2403 (3)	103

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

C(1)—N(2)	1·372 (6)	C(4a)—C(8a)	1·415 (7)
C(1)—C(8b)	1·378 (6)	C(5)—C(6)	1·375 (8)
N(2)—C(3)	1·383 (6)	C(6)—C(7)	1·377 (8)
N(2)—C(9)	1·486 (6)	C(7)—C(8)	1·396 (7)
C(3)—C(3a)	1·362 (7)	C(8)—C(8a)	1·389 (7)
C(3a)—N(4)	1·394 (6)	C(8a)—C(8b)	1·435 (6)
C(3a)—C(8b)	1·408 (6)	C(9)—C(10)	1·510 (8)
N(4)—C(4a)	1·387 (7)	C(9)—C(11)	1·513 (7)
N(4)—C(13)	1·443 (7)	C(9)—C(12)	1·495 (7)
C(4a)—C(5)	1·385 (8)		

C(1)—N(2)—C(9)	124·0 (4)	C(6)—C(7)—C(8)	120·8 (5)
C(1)—N(2)—C(3)	110·9 (4)	C(7)—C(8)—C(8a)	119·2 (5)
C(3)—N(2)—C(9)	124·6 (4)	C(4a)—C(8a)—C(8)	118·3 (4)
N(2)—C(3)—C(3a)	105·0 (4)	C(8)—C(8a)—C(8b)	135·0 (4)
C(3)—C(3a)—C(8b)	110·7 (4)	C(4a)—C(8a)—C(8b)	106·6 (4)
C(3)—C(3a)—N(4)	139·4 (4)	C(3a)—C(8b)—C(8a)	106·5 (4)
N(4)—C(3a)—C(8b)	109·9 (4)	C(1)—C(8b)—C(8a)	147·4 (4)
C(3a)—N(4)—C(13)	124·6 (4)	C(1)—C(8b)—C(3a)	106·0 (4)
C(3a)—N(4)—C(4a)	107·3 (4)	N(2)—C(9)—C(12)	111·0 (4)
C(4a)—N(4)—C(13)	128·1 (4)	N(2)—C(9)—C(11)	108·3 (4)
N(4)—C(4a)—C(8a)	109·7 (4)	N(2)—C(9)—C(10)	108·9 (4)
N(4)—C(4a)—C(5)	127·7 (5)	C(11)—C(9)—C(12)	111·3 (4)
C(5)—C(4a)—C(8a)	122·5 (5)	C(10)—C(9)—C(12)	108·2 (5)
C(4a)—C(5)—C(6)	117·4 (5)	C(10)—C(9)—C(11)	109·1 (5)
C(5)—C(6)—C(7)	121·9 (5)		

N(2)—C(3)—C(3a)—N(4)	179·8 (5)	N(4)—C(4a)—C(8a)—C(8)	178·1 (4)
N(4)—C(3a)—C(8b)—C(1)	−179·4 (4)	N(4)—C(4a)—C(5)—C(6)	−176·7 (5)
N(4)—C(3a)—C(8b)—C(8a)	−1·7 (5)	C(7)—C(8)—C(8a)—C(8b)	175·3 (5)
C(3a)—N(4)—C(4a)—C(5)	174·9 (5)	C(8)—C(8a)—C(8b)—C(1)	−0·4 (12)
N(4)—C(4a)—C(8a)—C(8b)	0·3 (5)		

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51056 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

N atom. There are no interactions between the molecules exceeding van der Waals forces.

Related literature. Kreher & Dyker (1987).

References

FRENZ, B. A. (1985). *Enraf–Nonius Structure Determination Package* (*SDP-PLUS*, V3.0). Enraf–Nonius, Delft, The Netherlands.

HEITKAMP, D. (1988). Dissertation, Univ. Dortmund, Federal Republic of Germany.

International Tables for X-ray Crystallography (1974). Vol. IV, Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

KELLER, E. (1986). *SCHAKAL. A Fortran Program for the Graphic Representation of Molecular and Crystallographic Models*. Univ. Freiburg, Federal Republic of Germany.

KREHER, R. P. & DYKER, G. (1987). *Z. Naturforsch. Teil B*, **42**, 473–477.

NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.

SHELDRICK, G. M. (1987). *SHELXTL PLUS* (Release 2) for Nicolet *R*3*m*/*V*. *Crystallographic Systems for Solving, Refining, and Displaying Crystal Structures from Diffraction Data*. Univ. of Göttingen, Federal Republic of Germany.

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Structure of (2*S*,3*R*)-3-Amino-2-phenylthiobutanoic Acid

BY HIROSHI NAKAI

Shionogi Research Laboratories, Shionogi & Co. Ltd, Fukushima-ku, Osaka 553, *Japan*

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Abstract. $C_{10}H_{13}NO_2S$, $M_r = 211{\cdot}28$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}723\ (2)$, $b = 19{\cdot}337\ (4)$, $c = 6{\cdot}365\ (2)$ Å, $V = 1073{\cdot}6\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}307$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu(\text{Mo } K\alpha) = 0{\cdot}27$ mm^{-1}, $F(000) = 448$, $T = 295$ K, $R = 0{\cdot}032$ for 1147 observed reflections [$F_o > 3\sigma(F_o)$]. The absolute configuration of C(2) was determined as *S* based on the *R* configuration of C(3) which was already known. The molecule adopts the zwitterion form with $-COO^-$ and $-NH_3^+$ groups. The H atoms in $-NH_3^+$ form hydrogen bonds with the O atoms in the $-COO^-$ group; (N4)H″···O(7) $(x, y, z) = 1{\cdot}90\ (3)$ [N···O 2·700 (3)], (N4)H···O(7) $(x-\frac{1}{2}, \frac{3}{2}-y, 1-z) = 1{\cdot}94\ (3)$ [2·818 (3)] and (N4)H′···O(8) $(x, y, 1+z) = 1{\cdot}97\ (3)$ Å [2·788 (3) Å].

Experimental. Prismatic colorless crystals obtained from methanol–water. Crystal of dimensions 0·2 × 0·2 × 0·5 mm. Rigaku AFC-5R diffractometer, graphite-monochromatized Mo *K*α radiation. Cell dimensions determined from 2θ angles in the range $15 < 2\theta < 24°$. Intensities measured up to $2\theta = 52°$ in *h* 0/10, *k* 0/23 and *l* 0/7, ω–2θ scans, ω-scan width $(1{\cdot}0 + 0{\cdot}45\tan\theta)°$, three standard reflections monitored every 100 measurements showed no significant change. 1214 unique reflections measured, 1147 intensities observed [$F_o \leq 3\sigma(F_o)$ and four very strong reflections rejected], no absorption corrections. Structures solved by *MULTAN*84 (Main, Germain & Woolfson, 1984). H atoms located on a difference density map. Positional parameters of all atoms and anisotropic thermal parameters of non-H atoms refined by block-diagonal

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

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
S(1)	0·5214 (1)	0·60192 (3)	0·1716 (1)	3·56 (1)
C(2)	0·5626 (2)	0·6748 (1)	0·3441 (3)	2·62 (4)
C(3)	0·5744 (2)	0·6517 (1)	0·5753 (3)	2·91 (4)
N(4)	0·5791 (2)	0·7155 (1)	0·7083 (2)	3·16 (4)
C(5)	0·7121 (3)	0·6059 (1)	0·6227 (3)	4·27 (6)
C(6)	0·7075 (2)	0·7132 (1)	0·2655 (3)	2·76 (4)
O(7)	0·7745 (2)	0·7503 (1)	0·3967 (2)	4·66 (4)
O(8)	0·7469 (2)	0·7067 (1)	0·0802 (2)	3·48 (3)
C(9)	0·3183 (2)	0·5951 (1)	0·1901 (3)	2·91 (4)
C(10)	0·2474 (3)	0·5569 (1)	0·3421 (4)	4·36 (6)
C(11)	0·0889 (3)	0·5503 (1)	0·3433 (5)	5·31 (7)
C(12)	0·0025 (3)	0·5812 (1)	0·1912 (5)	4·66 (6)
C(13)	0·0720 (3)	0·6196 (1)	0·0377 (5)	4·67 (6)
C(14)	0·2301 (3)	0·6268 (1)	0·0357 (4)	4·03 (5)

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

S(1)—C(2)	1·822 (2)	C(6)—O(8)	1·235 (3)
S(1)—C(9)	1·780 (2)	C(9)—C(10)	1·365 (3)
C(2)—C(3)	1·541 (3)	C(9)—C(14)	1·390 (3)
C(2)—C(6)	1·549 (3)	C(10)—C(11)	1·388 (4)
C(3)—N(4)	1·497 (3)	C(11)—C(12)	1·365 (5)
C(3)—C(5)	1·523 (3)	C(12)—C(13)	1·369 (5)
C(6)—O(7)	1·246 (3)	C(13)—C(14)	1·386 (4)

C(2)—S(1)—C(9)	102·3 (1)	O(7)—C(6)—O(8)	124·6 (2)
S(1)—C(2)—C(3)	111·4 (1)	S(1)—C(9)—C(10)	122·5 (2)
S(1)—C(2)—C(6)	109·7 (1)	S(1)—C(9)—C(14)	118·1 (2)
C(3)—C(2)—C(6)	113·1 (2)	C(10)—C(9)—C(14)	119·3 (2)
C(2)—C(3)—N(4)	107·6 (2)	C(9)—C(10)—C(11)	120·3 (2)
C(2)—C(3)—C(5)	114·2 (2)	C(10)—C(11)—C(12)	120·4 (3)
N(4)—C(3)—C(5)	110·2 (2)	C(11)—C(12)—C(13)	120·0 (3)
C(2)—C(6)—O(7)	116·2 (2)	C(12)—C(13)—C(14)	120·1 (3)
C(2)—C(6)—O(8)	119·1 (2)	C(9)—C(14)—C(13)	120·0 (2)

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