

The phenolic O and H(1) atoms lie close to the phenyl plane [0·003 (2) and 0·002 (27) Å], with the imino N atom at a distance of -0·151 (2) Å from the plane. The molecule as a whole is non-planar, the dihedral angle between the two rings is 82·5 (3) Å.

Torsion angles defining the relationship of the ethylideneamino group to the two rings are in Table 2. The imino N atom is *cis* to the thiophene S atom; the methyl C(6) atom is *trans* to the S atom and *cis* to the phenyl ring about the C=N bond.

The crystal structure is stabilized by an intermolecular O—H···N interaction with O···N = 2·835 (3), H···N = 2·08 (3) Å and an O—H···N angle of 160 (3)° with N at $\frac{1}{2} - x, -y, z$. There is also an intramolecular interaction of the same type with geometry (in order given above) of 2·866 (3), 2·53 (3) Å, and 108 (3)°.

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Structures of 5-Dimethylamino-1-[*N*-(2,6-dimethylphenyl)benzimidoyl]tetrazole, C₁₈H₂₀N₆, and 7,9-Dimethyl-5-dimethylamino-2-phenyl-3*H*-1,3,4-benzotriazepine, C₁₈H₂₀N₄

BY PETER F. LINDLEY

Department of Crystallography, Birkbeck College, Malet St, London WC1E 7HX, England

AND GERHARD V. BOYD AND GEORGE A. NICOLAOU

Department of Chemistry, King's College, Strand, London WC2R 2LS, England

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Abstract. (IVa), C₁₈H₂₀N₆, *M_r* = 320·40, monoclinic, *P*2₁/*c*, *a* = 9·785 (1), *b* = 7·701 (1), *c* = 23·847 (3) Å, β = 100·31 (1)°, *V* = 1768·0 (4) Å³, *Z* = 4, *D_x* = 1·203 g cm⁻³, λ(Cu *Kα*) = 1·54178 Å, μ = 6·14 cm⁻¹, *F*(000) = 680, *T* = 291 K, final *R* = 0·060 for 3091 independent reflections. The geometry of the five-membered ring resembles that of other tetrazoles. Thus the ring is planar and the N2—N3 linkage, 1·278 (4) Å, is predominantly double bond in character. (VIIb), C₁₈H₂₀N₄, *M_r* = 292·38, monoclinic, *P*2₁/*c*, *a* = 11·592 (1), *b* = 14·781 (2), *c* = 9·497 (1) Å, β = 98·32 (1)°, *V* = 1610·1 (3) Å³, *Z* = 4, *D_x* = 1·206 g cm⁻³, λ(Cu *Kα*) = 1·54178 Å, μ = 5·88 cm⁻¹, *F*(000) = 624, *T* = 291 K, final *R* = 0·056 for 2703 significant independent reflections. The seven-membered ring is severely puckered and the endocyclic bond lengths indicate double bonds

between N1—C2, 1·274 (3) Å, and N4—C5, 1·286 (2) Å. In the crystal the H atom at N3 is hydrogen bonded to N1 in a symmetry-related molecule; the N···N separation is 3·096 (3) Å.

Introduction. We recently reported general syntheses of 1- and 2-benzimidoyltetrazoles and the conversion of some of these compounds into novel 3*H*-1,3,4-benzotriazepines (Boyd, Cobb, Lindley, Mitchell & Nicolaou, 1987). Here we describe the X-ray structures of a tetrazole and a benzotriazepine.

Treatment of a mixture of *N*-benzoyl-2,6-dimethylaniline and dimethylcyanamide with phosphorus oxychloride, followed by perchloric acid, gave the salt (Ia) (Boyd, Khurshid, Nicolaou & Smith, 1988), which afforded the azido perchlorate (IIa) with sodium azide. The action of sodium

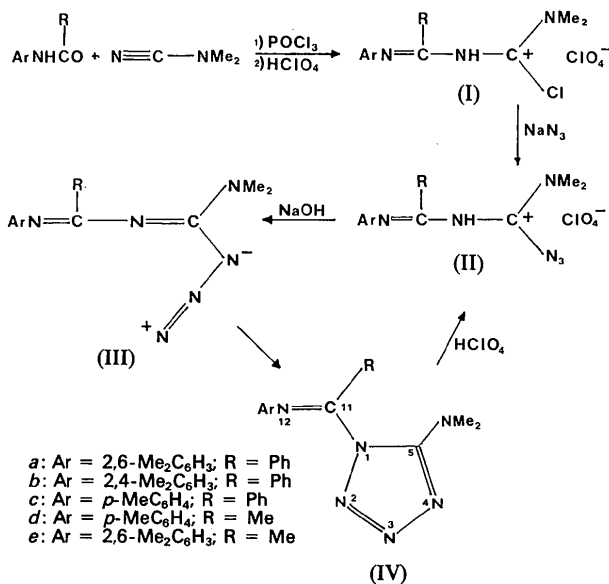
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Table 1. Crystal and refinement data for (IVa) and (VIIb)

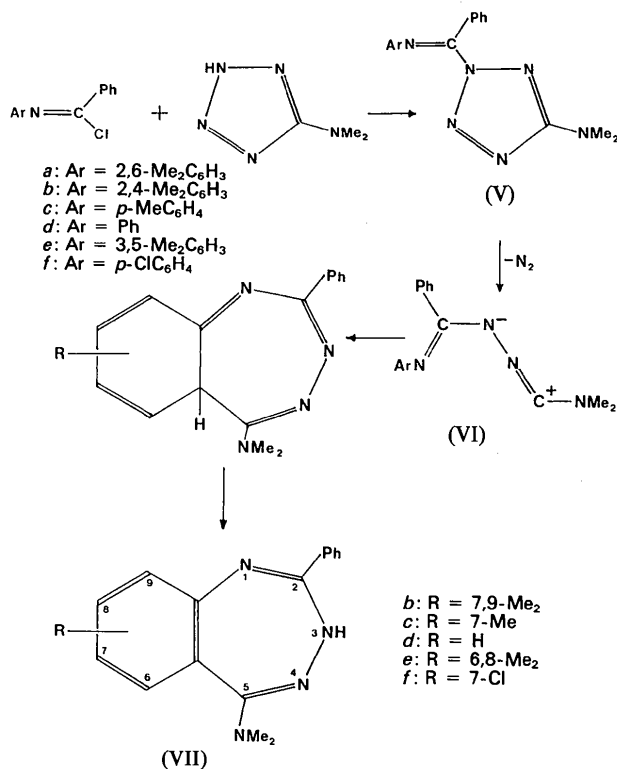
	(IVa)	(VIIb)
Crystal shape and size (mm)	Tabular on [001] 0.5 × 0.5 × 0.25	Prismatic 0.5 × 0.5 × 1.0
Reflections used for cell-parameter refinement: number and θ range (°)	21 25.2 ≤ θ ≤ 29.8	20 25.1 ≤ θ ≤ 29.7
Data-collection method	$\omega/2\theta$	$\omega/2\theta$
2 θ range (°)	1 < 2 θ < 140	1 < 2 θ < 140
Scan parameters: method, number of steps, step size (°)	Stationary background (70 + 0.142tan θ) 0.01	Moving window 120 (90) 0.01
Range of h	0 → 11	-13 → 13
k	-8 → 5	-18 → 0
l	-28 → 28	-7 → 10
Standard reflections: number, measuring frequency (reflections), maximum variation (%), overall radiation decay	4 50 9.6 Insignificant	4 50 2.1 Insignificant
Transmission-factor range	1.00–1.07	1.00–1.21
Number of reflections measured	4604	6089
Number of unique reflections	3091	2912
R_{int}	0.019	0.009
Number of reflections used in refinement	3091	2703
Omission criteria		$F < 5\sigma(F)$
Number of parameters refined	298	200
Final R value	0.060	0.056
$w = 1/[\sigma^2(F) + AF^2]$; A	0.000296	0.000385
Isotropic extinction parameter, X	0.24 (1) × 10 ⁻³	0.33 (2) × 10 ⁻³
Final wR	0.090	0.072
Final largest shift/e.s.d.	0.91	0.48
Max. and min. heights in final difference Fourier map (e Å ⁻³)	0.31, -0.27	0.26, -0.22

hydroxide on the azido salt gave the tetrazole (IVa) by spontaneous 1,5-dipolar cyclization of the azide (IIIa). Treatment of the tetrazole with perchloric acid regenerated the azido perchlorate (IIa). Since the compound (IVa) represents the first authentic 1-imidoyltetrazole, its X-ray structure was determined.



The 1-benzimidoyltetrazole (IVb) was similarly prepared, starting with *N*-benzoyl-2,4-dimethylaniline. The 2-benzimidoyl isomer (Vb) was obtained by the reaction of 5-dimethylaminotetrazole with *N*-(2,4-dimethylphenyl)benzimidoyl chloride in pyri-

dine. Both tetrazoles lost nitrogen on heating to give the benzotriazepine (VIIb), whose X-ray structure was determined. This is the first fully unsaturated member of this class without a substituent on nitrogen (Richter & Morgenstern, 1984). We propose that the 1-benzimidoyl isomer (IVb) rearranges by a symmetry-allowed [1,5] shift of the benzimidoyl group to the 2-substituted tetrazole (Vb) and that the latter decomposes to the nitrile imine (VIb) (Huisgen, Sauer & Seidel, 1960), which forms the product by an antarafacial 1,7-dipolar cyclization, followed by a [1,5]-*H* shift.



A number of analogous reactions were carried out; these are summarized in the reaction schemes above.

Experimental. Preparative and analytical details for the title compounds and the other relevant compounds involved in the reaction scheme are described in a supplementary publication.* X-ray intensity data for (IVa) and (VIIb) were collected on a Hilger and Watts Y290 four-circle diffractometer with Ni-

* Complete preparative and analytical details for the compounds involved in the reaction schemes, together with lists of structure factors, anisotropic temperature factors and H-atom parameters, and details of molecular geometries for (IVa) and (VIIb) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52879 (34 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

filtered Cu $K\alpha$ radiation; the principal data collection and refinement parameters are listed in Table 1. In the case of (IVa), data were collected using the moving-window technique (Tickle, 1975); each scan consisted of some 120 steps of which 90 were taken to represent the peak and the remainder background. For (VIIb) stationary background counts were taken at both sides of each scan for one-tenth of the total scan time and the scan width was expanded in an integral number of steps to allow for dispersion. For both compounds intensity data were corrected for Lorentz and polarization effects and absorption corrections were made using the empirical method of North, Phillips & Mathews (1968).

The structures were solved using the direct methods program *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and refinement on F^2 s was carried out with the full-matrix program *SHELX76* (Sheldrick, 1976). Initially all non-H atoms were treated isotropically and most H atoms then located as diffuse electron maxima, typically $0.3\text{--}0.6 e \text{ \AA}^{-3}$, from difference Fourier syntheses. In subsequent refinement the H atoms were placed in idealized positions assuming C—H and N—H bond distances of 1.09 and 1.00 Å respectively, and held constant; the non-H atoms were permitted to refine anisotropically. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$ and refinement was terminated when the parameter shifts were less than their corresponding standard deviations. In the case of both structures inspection of the low angle, high intensity reflections revealed systematic discrepancies between $|F_o|$ and $|F_c|$ and in the final cycles an empirical isotropic extinction parameter, X , was included using the expression: $F_c' = F_c [1 - (XF_c^2/\sin\theta)]$. Atomic scattering factors were taken from Cromer & Mann (1968). Molecular-geometry calculations were performed using 'in-house' programs (Lindley, 1985).

Discussion. Refined atomic coordinates and equivalent isotropic thermal parameters for (IVa) and (VIIb) are given in Table 2,* bond lengths and angles are listed in Table 3. The atomic numbering schemes adopted are implicit in the compound names and indicated in the reaction schemes. Figs. 1(a) and 1(b) are stereo drawings of the molecules of (IVa) and (VIIb).

In (IVa) the tetrazole moiety is planar with the largest deviation out of the plane defined by atoms N1—C5 inclusive, being $-0.009(3) \text{ \AA}$ for N2. The N2—N3 interaction, $1.278(4) \text{ \AA}$ has clear double-bond character, but the remaining endocyclic bond lengths suggest substantial electron delocalization over N1, N4 and C5. N51 may be involved to some

Table 2. Fractional coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\times 10^3$) for (IVa) and (VIIb)

	x	y	z	U_{eq}^*
(IVa)				
N1	6706 (2)	-593 (2)	807 (1)	39 (1)
N2	6515 (2)	-2052 (2)	470 (1)	48 (1)
N3	6517 (2)	-1544 (2)	-40 (1)	51 (1)
N4	6666 (2)	207 (2)	-71 (1)	50 (1)
C5	6777 (2)	787 (2)	460 (1)	42 (1)
C11	7084 (2)	-868 (2)	1417 (1)	41 (1)
N12	8171 (2)	-270 (2)	1724 (1)	47 (1)
C111	6094 (2)	-1966 (2)	1656 (1)	43 (1)
C112	4691 (2)	-2068 (3)	1401 (1)	53 (1)
C113	3801 (2)	-3062 (3)	1654 (1)	70 (1)
C114	4260 (3)	-3980 (4)	2142 (1)	76 (1)
C115	5643 (3)	-3902 (3)	2392 (1)	72 (1)
C116	6562 (2)	-2898 (3)	2154 (1)	55 (1)
C121	9233 (2)	682 (3)	1529 (1)	50 (1)
C122	9591 (2)	2288 (3)	1788 (1)	59 (1)
C123	10640 (3)	3249 (4)	1615 (1)	86 (1)
C124	11360 (3)	2593 (6)	1213 (2)	106 (2)
C125	11061 (3)	963 (6)	989 (1)	94 (1)
C126	9999 (2)	-43 (4)	1143 (1)	65 (1)
C127	8895 (3)	2945 (4)	2261 (1)	73 (1)
C128	9781 (3)	-1856 (4)	910 (1)	88 (1)
N51	6924 (2)	2465 (2)	620 (1)	57 (1)
C52	7143 (4)	3697 (4)	177 (1)	90 (1)
C53	6163 (3)	3137 (3)	1043 (1)	72 (1)
(VIIb)				
N1	5351 (1)	1622 (1)	3566 (2)	43 (1)
C2	5727 (1)	2129 (1)	4617 (2)	42 (1)
N3	5055 (1)	2386 (1)	5665 (2)	48 (1)
N4	4096 (1)	2968 (1)	5136 (2)	45 (1)
C5	3275 (1)	2587 (1)	4284 (2)	41 (1)
C5a	3246 (1)	1629 (1)	3825 (2)	42 (1)
C6	2197 (2)	1157 (1)	3702 (2)	50 (1)
C7	2116 (2)	256 (1)	3278 (2)	53 (1)
C8	3120 (2)	-162 (1)	2956 (2)	54 (1)
C9	4179 (2)	292 (1)	3036 (2)	49 (1)
C9a	4245 (2)	1194 (1)	3504 (2)	42 (1)
C21	6956 (2)	2448 (1)	4772 (2)	49 (1)
C22	7773 (2)	1926 (2)	4207 (3)	76 (1)
C23	8915 (2)	2212 (3)	4311 (4)	100 (1)
C24	9262 (2)	3004 (2)	4987 (4)	86 (1)
C25	8462 (2)	3519 (2)	5537 (4)	93 (1)
C26	7302 (2)	3249 (2)	5441 (3)	72 (1)
N51	2282 (1)	3104 (1)	3844 (2)	47 (1)
C52	2258 (2)	3997 (1)	4502 (3)	57 (1)
C53	1869 (2)	3130 (2)	2314 (2)	65 (1)
C71	963 (2)	-232 (2)	3112 (3)	78 (1)
C91	5235 (2)	-180 (2)	2625 (3)	69 (1)

$$*U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

extent in this electron delocalization although the geometry at N51 is not planar. N51 is displaced by $-0.245(2) \text{ \AA}$ from the plane through C5, C52 and C53, and the dimethylamino moiety is considerably twisted with respect to the tetrazole ring owing to steric hindrance between the methyl groups at C53 and C127 [non-bonded separation $3.581(4) \text{ \AA}$]. The dihedral angle between the tetrazole ring and the plane through C5, C52 and C53 is $-25.97(9)^\circ$. The geometry in the benzimidoyl moiety is also nearly planar at C11 as would be expected with the presence of the double bond at C11—N12, $1.265(3) \text{ \AA}$, with C11 lying $-0.011(2) \text{ \AA}$ out of the least-squares plane defined by N1, C11, N12 and C111; however, the dihedral angle between this plane and that of the tetrazole ring is $52.25(9)^\circ$. The phenyl ring of the benzimidoyl group is planar within experimental error and is twisted by $26.41(7)^\circ$ about the

* See deposition footnote.

Table 3. Bond distances (Å) and bond angles (°) for the non-H atoms in (IVa) and (VIIb); *e.s.d.*'s include cell parameter *e.s.d.*'s

(IVa)			
N1—N2	1.375 (3)	C113—C114	1.367 (4)
N1—C5	1.356 (3)	C114—C115	1.380 (4)
N1—C11	1.451 (4)	C115—C116	1.384 (4)
N2—N3	1.278 (4)	C121—C122	1.399 (3)
N3—N4	1.360 (2)	C121—C126	1.403 (4)
N4—C5	1.329 (4)	C122—C123	1.387 (4)
C5—N51	1.348 (2)	C122—C127	1.506 (4)
C11—N12	1.265 (3)	C123—C124	1.383 (5)
C11—C111	1.475 (3)	C124—C125	1.375 (6)
N12—C121	1.417 (3)	C125—C126	1.397 (4)
C111—C112	1.401 (3)	C126—C128	1.504 (4)
C111—C116	1.393 (3)	N51—C52	1.464 (4)
C112—C113	1.377 (3)	N51—C53	1.452 (4)
N2—N1—C5	107.5 (2)	C113—C114—C115	119.6 (3)
N2—N1—C11	116.8 (2)	C114—C115—C116	120.3 (2)
C5—N1—C11	134.0 (2)	C111—C116—C115	120.0 (2)
N1—N2—N3	106.4 (2)	N12—C121—C122	116.9 (2)
N2—N3—N4	111.9 (2)	N12—C121—C126	121.3 (2)
N3—N4—C5	105.8 (2)	C122—C121—C126	121.2 (2)
N1—C5—N4	108.3 (2)	C121—C122—C123	118.7 (2)
N1—C5—N51	126.4 (2)	C121—C122—C127	121.1 (2)
N4—C5—N51	125.3 (2)	C123—C122—C127	120.1 (2)
N1—C11—C12	124.3 (2)	C122—C123—C124	120.5 (3)
N1—C11—C111	113.8 (2)	C123—C124—C125	120.2 (4)
N12—C11—C111	121.8 (2)	C124—C125—C126	121.4 (3)
C11—N12—C121	126.1 (2)	C121—C126—C125	117.5 (3)
C11—C111—C112	121.7 (2)	C121—C126—C128	123.7 (2)
C11—C111—C116	119.1 (2)	C125—C126—C128	118.7 (3)
C112—C111—C116	119.2 (2)	C5—N51—C52	116.0 (2)
C111—C112—C113	119.2 (2)	C5—N51—C53	119.8 (2)
C112—C113—C114	121.6 (2)	C52—N51—C53	115.5 (2)
(IVb)			
N1—C2	1.274 (3)	C7—C71	1.507 (3)
N1—C9a	1.423 (3)	C8—C9	1.391 (3)
C2—N3	1.402 (3)	C9—C9a	1.404 (2)
C2—C21	1.488 (3)	C9—C91	1.509 (3)
N3—N4	1.438 (2)	C21—C22	1.388 (3)
N4—C5	1.286 (2)	C21—C26	1.376 (3)
C5—C5a	1.481 (2)	C22—C23	1.380 (4)
C5—N51	1.394 (2)	C23—C24	1.367 (5)
C5a—C6	1.392 (2)	C24—C25	1.361 (4)
C5a—C9a	1.396 (3)	C25—C26	1.393 (4)
C6—C7	1.391 (2)	N51—C52	1.462 (3)
C7—C8	1.390 (3)	N51—C53	1.463 (3)
C2—N1—C9a	119.9 (2)	C8—C9—C9a	118.9 (2)
N1—C2—N3	123.6 (1)	C8—C9—C91	120.4 (2)
N1—C2—C21	118.6 (2)	C9a—C9—C91	120.8 (2)
N3—C2—C21	117.8 (2)	N1—C9a—C9	123.8 (1)
C2—N3—N4	113.3 (2)	N1—C9a—C91	116.3 (2)
N3—N4—C5	115.0 (1)	C5a—C9a—C9	119.8 (2)
N4—C5—C5a	126.0 (1)	C2—C21—C22	118.9 (2)
N4—C5—N51	116.9 (2)	C2—C21—C26	122.0 (2)
C5a—C5—N51	117.0 (1)	C22—C21—C26	119.2 (2)
C5—C5a—C6	119.1 (1)	C21—C22—C23	120.2 (3)
C5—C5a—C9a	121.4 (1)	C22—C23—C24	120.7 (3)
C6—C5a—C9a	119.5 (2)	C23—C24—C25	119.2 (2)
C5a—C6—C7	121.7 (2)	C24—C25—C26	121.3 (3)
C6—C7—C8	117.8 (2)	C21—C26—C25	119.4 (3)
C6—C7—C71	120.7 (2)	C5—N51—C52	115.6 (1)
C8—C7—C71	121.5 (2)	C5—N51—C53	116.7 (2)
C7—C8—C9	122.3 (1)	C52—N51—C53	112.1 (2)

C11—C111 bond to minimize non-bonded contacts between the *ortho*-H atom and the tetrazole ring [closest approach, N1 to H112, 2.549 (2) Å]. The second phenyl ring, C121—C126 inclusive, is not strictly planar with maximum displacements of -0.030 (3) and 0.025 (3) Å for C121 and C122 respectively. The methyl substituents at the 2 and 6 positions are displaced by 0.166 (5) and 0.142 (5) Å from the ring plane and the geometrical distortions in this region of the molecule are presumably due to

steric hindrance; C127 is close to C53, *vide ultra*, whereas C128 lies above the plane of the tetrazole ring with a closest non-bonded N2...C128 separation of 3.184 (4) Å. In the crystal structure, Fig. 2(a), the molecules are arranged in layers running approximately parallel to *a** and there are no non-bonded contacts significantly closer than the sum of the respective van der Waals radii.

For compound (VIIb) a planar seven-membered triazepine ring would imply anti-aromaticity, since the ring atoms possess eight π electrons; in the crystal structure this ring is highly puckered with hinges at N1—C5 and C2—N4. Thus, the displacements of C2 and N4 out of the least-squares plane defined by N1, C5, C5a and C9a are

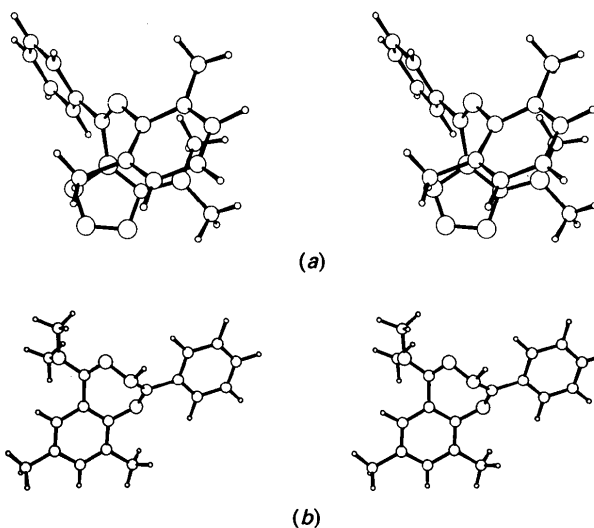


Fig. 1. Stereo drawings of the molecules of (a) (IVa) viewed along the *a* axis and (b) (VIIb) viewed along the *c* axis.

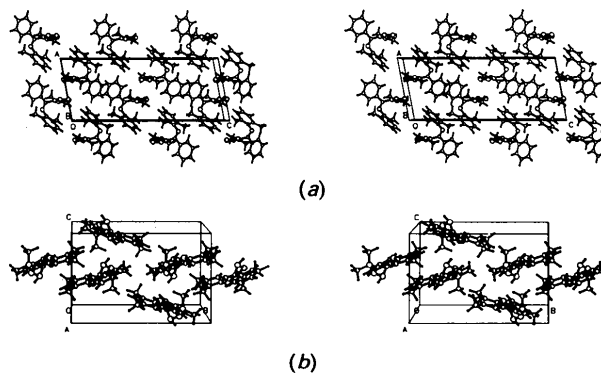


Fig. 2. Stereo drawings of the molecular packings of (a) (IVa) viewed along the *b* axis, showing layers of molecules parallel to *a**, and (b) (VIIb) viewed along the *a** axis. Molecules related by the *c* glide plane form intermolecular hydrogen bonds, 3.096 (3) Å, between pairs of N1 and N3 atoms as follows: N1...N3(*x*, $\frac{1}{2} - y$, $-\frac{1}{2} + z$) and N3...N1(*x*, $\frac{1}{2} - y$, $\frac{1}{2} + z$).

−0.754 (4) and −0.683 (4) Å respectively whereas N3 is displaced by −1.499 (3) Å out of this plane. The endocyclic bond lengths clearly indicate double bonds at N1—C2, 1.274 (3), and N4—C5, 1.286 (2) Å. In the dimethylamino moiety the N51 atom has essentially sp^3 -hybridized character [N51 lies some −0.332 (2) Å out of the plane through C5, C52 and C53], and the C5—N51 bond length, 1.394 (2) Å, is appreciably longer than the corresponding distance in (IVa). Steric hindrance is minimized by the dimethylamino moiety twisting away from the main plane through the molecule; the dihedral angle between the planes through C5, C5a, C9a and N1, and C5, C52 and C53 is 65.44 (1)°. The phenyl ring at C2 is planar within experimental error and makes a dihedral angle of 28.2 (1)° with the plane defined by N1, C2 and N3 in order to minimize the non-bonded contact between the H atom bonded to N3 and the *ortho*-H atom on the phenyl ring. The remaining aromatic ring, atoms C5a, C6, C7, C8, C9 and C9a, is not strictly planar, the largest deviation being 0.014 (2) Å for C9; the displacements of the 7- and 9-methyl substituents are 0.046 (4) and 0.062 (4) Å respectively. In the crystal structure, Fig. 2(b), the molecules pack so that a network of intermolecular hydrogen bonds is formed between pairs of N1 and N3 atoms, *viz.* N1⋯N3($x, \frac{1}{2} - y, -\frac{1}{2} + z$) and N3⋯N1($x, \frac{1}{2} - y, \frac{1}{2} + z$), 3.096 (3) Å.

All the figures were drawn using *PLUTO78* (Motherwell & Clegg, 1978), as modified by Dr A. Quick to run on the Amdahl computer at the University of London Computer Centre.

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Structure of Ethyl 5-Ethoxy-3-phenyl-2-indolecarboxylate

BY T. N. CHANDRAKANTHA AND PUTTARAJA

Department of Physics, Jnana Bharathi Campus, Bangalore University, Bangalore-560 056, India

AND VASANTHA PATTABHI

Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Madras-600 025, India

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Abstract. $C_{19}H_{19}NO_3$, $M_r = 309.37$, monoclinic, $P2_1/c$, $a = 10.322$ (3), $b = 21.063$ (5), $c = 15.006$ (5) Å, $\beta = 92.93$ (3)°, $V = 3258.22$ (1) Å³, $Z = 8$, $D_m = 1.27$ (5), $D_x = 1.26$ Mg m⁻³, $\lambda(\text{Cu K}\alpha) = 1.5418$ Å, $\mu = 0.61$ mm⁻¹, $F(000) = 1312$, $T = 300$ K, final $R = 0.048$ for 4361 observed reflections. Two independent molecules per asymmetric unit. The phenyl ring connected to the planar indole rings is rotated out of the indole plane by 44.7 (4)° in molecule *A* and 47.3 (5)° in molecule *B*. The ethoxy-

carbonyl group makes an angle of 4.4 (6)° with the mean plane of the indole rings in molecule *A* and 14.7 (5)° in molecule *B*. The molecules in the asymmetric unit are held together by N—H⋯O bonds.

Introduction. Indole derivatives are important pharmacologically, possessing anti-allergic, central nervous system depressant and muscle relaxant properties (Harris & Uhle, 1960; Wei & Stanley,