Reaction of Picryl Azide with *N*-Methylindoles: the Crystal Structure of 1-Methyl-2-picryliminoindoline

By **A. S. Bailey*** and (Mrs.) **W. A. Warr,** Dyson Perrins Laboratory, South Parks Road, Oxford
G. B. Allison and **C. K. Prout,** Chemical Crystallography Laboratory, South Parks Road, Oxford

Picryl azide reacts with *N*-methylindoles to form 2-*N*-picryl derivatives of 2-aminoindole. These compounds exist in the amino-form (I) in solution, but the three-dimensional crystal structure determination for the 1-methyl-2-*N*-picryl derivative shows it to be in the iminoindoline form (II). The structure was determined from diffractometer data by direct methods and refined by least squares to *R* 0·053. The carbon–carbon bonds at C(3) are both of length 1·52 Å, and the positions of the two hydrogen atoms were clearly established from difference Fourier syntheses.

2-ALKYLINDOLES react with picryl azide forming 3,3′-azoindoles and with toluene-*p*-sulphonyl azide to form a mixture of the azo-compound and the 3-*p*-tolylsulphonylaminoindole.[1] Simple indoles unsubstituted in the 2-position form 2-*p*-tolylsulphonylaminoindoles [(I), (II); R^3 = tosyl] when warmed with toluene-*p*-sulphonyl azide.[2,3] We now report the reaction of

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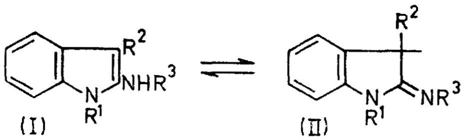
[1] A. S. Bailey and J. J. Merer, *J. Chem. Soc.* (*C*), 1966, 1345.

[2] A. S. Bailey, M. C. Churn, and J. J. Wedgwood, *Tetrahedron Letters*, 1968, 5953.

[3] A. S. Bailey and (Mrs.) W. A. Warr, unpublished observations.

picryl azide with *N*-substituted indoles, together with the crystal and molecular structure of one of the adducts, in order to check the spectral assignment.



(I) (II)

RESULTS AND DISCUSSION

Chemistry.—*N*-Methylindole, 1,3-dimethylindole, and indole-*N*-acetic acid react smoothly with picryl azide at room temperature. The products are sparingly soluble crystalline materials having similar u.v. spectra (Figure 1; the values of ε for curve A are probably too small, since the compound is very insoluble).

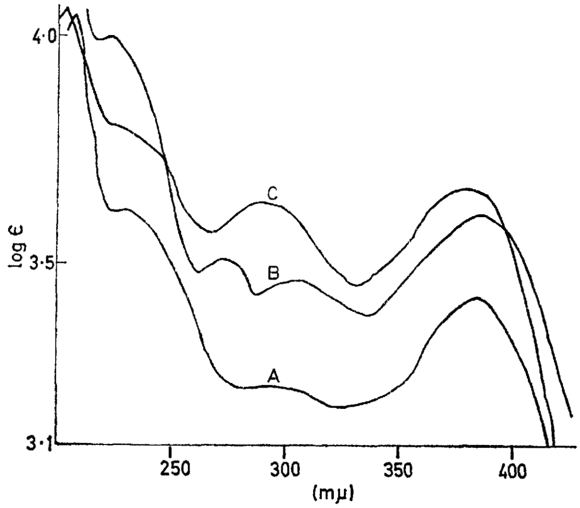


FIGURE 1 U.v. spectra of A, (I; $R^1 = Me$, $R^2 = H$, $R^3 =$ picryl); B, (I; $R^1 = R^2 = Me$, $R^3 =$ picryl); and C, (I; $= CH_2{\cdot}CO_2H$, $R^2 = H$, $R^3 =$ picryl), all in solution in ethanol

The mass spectra of these compounds gave little structural information. The base peak in the spectrum of (I; $R^1 = Me$, $R^2 = H$, $R^3 =$ picryl) appeared at *m/e* 339 ($M - H_2O$). The molecular ion signal *m/e* 357 (14%) was small. Main peaks in the higher mass region arise from loss of NO or NO_2: 309 (13%, 339 − NO), 293 (14%, 339 − NO_2), and 247 (30%, 293 − NO_2). The second most intense peak in the spectrum [*m/e* 224 (42%)] ($C_7H_4N_4O_5$) probably arises by cleavage of the indole ring. There were no strong signals in the region of *m/e* 144, suggesting that picryl cleavage is unimportant and no peaks at 227—229 or 131, suggesting that C-/-N-picryl cleavage is not occurring. A small signal at *m/e* 328 ($M - 29$) corresponds to loss of H and CO (less likely is 2H + HCN). This may indicate oxygen transfer to C(3) of the indole structure followed by loss of carbon monoxide (*cf*. 1-nitronaphthalene[4]). The third strongest signal, *m/e* 105 (30%, $PhCO^+$), may be formed in this manner.

The mass spectrum of (I; $R^1 = R^2 = Me$, $R^3 =$ picryl) contains a weak molecular ion *m/e* 371 (4%), 356 (10%, $M - CH_3$), 355 (40%, $M - O$), 341 (10%, $M - NO$), and 340 (34%, $M - H - NO$). The base peak is at *m/e* 337 (100%, $M - H - CH_3 - H_2O$) and the second most intense peak at *m/e* 312 (54%, 355 − CH_3 − CO). Other intense signals were formed by loss of NO or NO_2; 323 (23%, $M - H_2O - NO$), 307 (31%, $M - H_2O - NO_2$) 293 (22%, 323 − NO), 291 (18%, 337 − NO_2), 245 (22%, 337 − $2NO_2$), 266 (8%, 312 − NO_2), and 220 (13%, 266 − NO_2). There were no strong peaks below *m/e* 220. The following transformations were indicated by metastable peaks: 338 ⟶ 337 (336·0), 355 ⟶ 340 (325·6), 355 ⟶ 312 (274·5), 320 ⟶ 290 (262·8), 312 ⟶ 266 (226·6), and 266 ⟶ 220 (181·9).

The n.m.r. spectra ([$^2H_6$]dimethyl sulphoxide) of the three products all show the signals of the 'picryl' protons split into a pair of doublets with separations varying from 42 to 62 Hz and coupling constants of 2 Hz. The compound formed from 1-methylindole is rather insoluble in dimethyl sulphoxide and gives a poor spectrum; the spectra of the compound formed from 1,3-dimethylindole is shown (Figure 3). The splitting persists in trifluoracetic acid and, for the indole-*N*-acetic acid adduct, in deuterium oxide–sodium carbonate. The n.m.r. spectrum of *N*-phenylpicramide shows a sharp singlet [τ (Me_2SO) 1·1; τ ($CHCl_3$) 0·92] for the picryl protons.

The n.m.r. spectrum of 1,1-diphenyl-2-(2,4,6-trinitrophenyl)hydrazine showed the picryl signals as a broad band at 36° (methylene chloride) which had split into an AB system at −60°;[5] the signal from *N*-methylpicramide consisted of a sharp singlet at +38° and an AB system at −60°. The difference in chemical shifts for the hydrazine derivative varied with solvent, being smaller in acetone than in methylene chloride. A sample of (I; $R^1 = R^2 = Me$, $R^3 =$ picryl) was heated in a variable-temperature probe; the AB spectrum of the picryl protons persisted up to 160°; the compound then decomposed.

The spectral information suggested two structural possibilities for these compounds.

(i) That the adducts were oxidation–reduction products—alkylindoles are readily oxidised at the 3-position, and aromatic nitro-compounds oxidise enamines.[6]

(ii) That the adducts were as predicted [either (I) or (II)], with severe restriction of free rotation of the *o*-nitro-groups.

This latter effect has been observed before.[5] The restriction in rotation is presumably due to hydrogen bonding between an *o*-nitro-group and a hydrogen atom attached to the nitrogen. This hypothesis requires that in solution the adduct is present in the indole (I) rather

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[4] H. Budzikiewicz, C. Djerassi, and D. H. Williams, 'Mass Spectrometry of Organic Compounds,' Holden Day, London, 1967, 518.

[5] J. Heidberg, J. A. Weil, G. A. Janusonis, and J. K. Anderson, *J. Chem. Phys.*, 1964, **41**, 1033.

[6] S. Danishefsky and R. Cavanaugh, *Chem. and Ind.*, 1967, 2171.

than the indoline (II) form. Strong hydrogen bonding has been observed in the crystal structure of 2,3,4,6-tetranitroaniline[7] (O · · · H contacts of 1·92 and 2·04 Å). An accurate crystal structure analysis (R = 0·053) of the *N*-methylindole adduct showed that it is of type (II) and is in the indoline form (Figure 2).

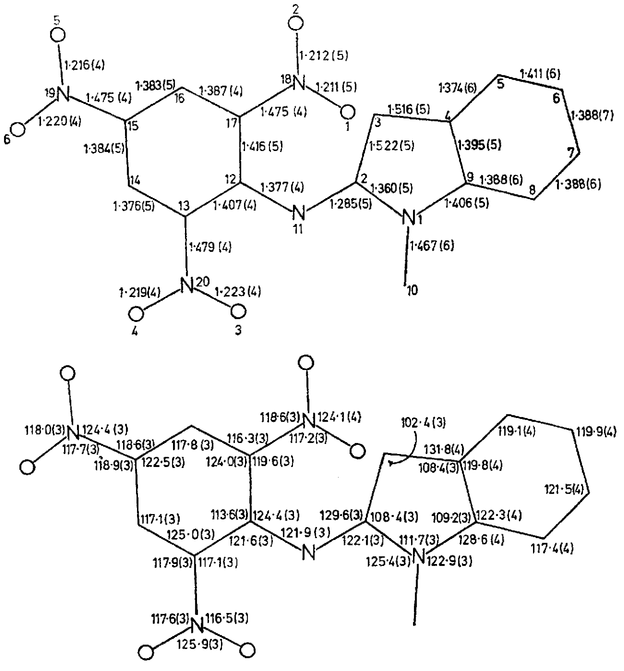

FIGURE 2 Bond distances (Å) and angles (°), together with their estimated standard deviations

The n.m.r. spectra (Figure 3) can be explained if, in solution (Me_2SO), the compound exists as the indole (I; R^1 = Me, R^2 = H, R^3 = picryl), as there is no sign of the signal to be expected from the methylene group in (II; R^1 = Me, R^2 = H, R^3 = picryl), although the corresponding tolylsulphonyl derivative (R^1 = Me, R^2 = H, R^3 = tosyl) is mainly (80%) in the imino-form (II).[3] The proton at C(3) in the picryl compound is deshielded by the picryl group and its signal is moved into the aromatic region. This contrasts with the signal from H–C(3) in (I; R^1 = Me, R^2 = H, R^3 = tosyl), which appears at τ 4·1.[3] A similar effect is seen in the 3-methyl-substituted compounds. In (I; R^1 = R^2 = Me, R^3 = tosyl) this signal (Me) appears at τ 8·38; when R^3 = picryl the signal is moved down to τ 7·48. The acid (R^1 = $CH_2{\cdot}CO_2H$, R^2 = H, R^3 = picryl) is entirely in form (I) in both dimethyl sulphoxide and deuterium oxide–sodium carbonate, and the signal from H–C(3) is in the aromatic region. The 1-methyl compound and the 1,3-dimethyl compound (R^3 = picryl) exist in the indole form (I) in trifluoracetic acid (see Figure 3), whereas the corresponding tosyl derivatives exist in form (II; R^3 = tosyl).

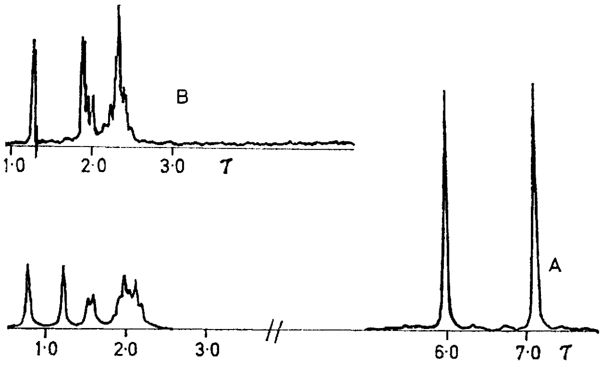

FIGURE 3 N.m.r. spectra of (I; R^1 = R^2 = Me, R^3 = picryl) in solution in A, trifluoroacetic acid and B, dimethyl sulphoxide

Crystal Structure of the N-*Methylindole Adduct*.—The atom numbering, interatomic distances, and interbond angles, together with their estimated standard deviations, are shown in Figure 2.

The compound is formulated as an indoline adduct on the basis of the following evidence. (i) On a difference Fourier map at the convergence of isotropic refinement (R 0·128), two clearly resolved peaks of *ca.* 0·7e at *ca.* 1 Å from C(3), completed a tetrahedron about this atom, and must correspond to hydrogen atom positions. The other nine hydrogen atom positions were equally well defined, but no maxima were observed about N(11). (ii) Table 1 gives a comparison of bond lengths reported for indole compounds with those obtained in this work. Of especial interest is the bond C(2)–C(3). In the known indole structures the length of this bond agrees well with the calculated value (1·34 Å):[10] the π-bond order is 0·5. Our bond length (1·522 Å) corresponds to that of a carbon–carbon (C–C=C) single σ-bond (1·510 Å),[11] as required by the indoline formulation. Other bond lengths in the heterocycle show the expected deviations from the indole values. (iii) On the basis of a semi-empirical

TABLE 1

Comparison of the dimensions (Å) of indole compounds

Bond	L-Tryptophan hydrochloride [a]	Indole-3-acetic acid [b]	This work	Calc. for indole [c]
N(1)–C(2)	1·377	1·401	1·360	1·36
C(2)–C(3)	1·344	1·342	1·522	1·36
C(3)–C(4)	1·451	1·470	1·516	1·42
C(4)–C(5)	1·412	1·434	1·374	1·41
C(5)–C(6)	1·397	1·409	1·411	1·38
C(6)–C(7)	1·386	1·396	1·388	1·40
C(7)–C(8)	1·399	1·409	1·388	1·38
C(8)–C(9)	1·400	1·422	1·388	1·40
C(4)–C(9)	1·382	1·407	1·395	1·41
N(1)–C(9)	1·391	1·385	1·406	1·37
σ (mean)	0·015	0·018	0·005	

[a] Ref. 8. [b] Ref. 9. [c] Ref. 10.

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[7] C. Dickinson, J. M. Stewart, and J. R. Holden, *Acta Cryst.*, 1966, **21**, 663.

[8] T. Takigawa, T. Ashida, Y. Sasada, and M. Kakudo, *Bull. Chem. Soc. Japan*, 1966, **39**, 2369.

[9] I. L. Karle, K. Butts, and P. Gum, *Acta Cryst.*, 1964, **17**, 496.

[10] H. C. Longuet-Higgins and C. A. Coulson, *Trans. Faraday Soc.*, 1947, **43**, 87.

[11] *Chem. Soc. Special Publ.*, No. 18, 1965.

bond-order–bond-length relationship,[12] the π-bond order of C(2)–N(11)(1·285 Å) is very nearly one. N(11)–C(12) is longer (1·377 Å), although not as long as expected for a single σ-bond, *e.g.* in a picryl azide molecular complex the corresponding bond length is 1·46 Å.[13] The indoline formulation requires double and single bonds in these respective positions. (iv) The indoline structure requires that N(11) should be sp^2 hybridised and that the hybrid orbitals should be coplanar with the indoline ring. C(12) is coplanar and N(11) is only slightly out of plane (0·05 Å) as can be seen from Table 2, which shows the equations of some least-squares planes through the molecule, together with deviations of atoms from the planes. For the indoline ring, no atom deviates by $>2\sigma$ from the plane, but the picryl ring is significantly non-planar (deviations $>3\sigma$). This effect is believed to be a real one, having been noticed before for polysubstituted benzene nuclei,[7] and is possibly due to a steric effect.

TABLE 2

Least-squares planes

(*a*) Distances (Å) of atoms from planes (parentheses indicate atoms not in calculation)

	Indoline	Picryl nucleus
N(1)	−0·002	
C(2)	0·007	
C(3)	−0·011	
C(4)	0·009	
C(5)	0·008	
C(6)	−0·002	
C(7)	−0·011	
C(8)	−0·005	
C(9)	−0·003	
C(10)	0·009	
C(11)	(0·055)	
C(12)	(−0·0002)	−0·002
C(13)		0·016
C(14)		−0·014
C(15)		−0·001
C(16)		0·014
C(17)		−0·013

(*b*) Equations of planes: $ax + by + cz = 1$ where x, y, and z are fractional co-ordinates

Plane	*a*	*b*	*c*
Indoline	−1·788	21·25	−37·46
Picryl nucleus	1·098	0·926	−0·381
Nitro-group on C(13)	−1·340	0·800	1·250
Nitro-group on C(15)	1·012	0·917	−0·340
Nitro-group on C(17)	0·186	1·101	−0·185

The angle between the least-squares planes through the indoline and picryl nuclei is 96°. The *o*-nitro-groups are twisted out of the plane of the picryl nucleus by 137 and 125°, while the *p*-nitro-group is almost coplanar with it. The direction of rotation is that suggested in ref. 14, *i.e.* right-handed about the direction of numbering.

The crystal lattice consists of isolated molecules held together by van der Waals forces. The picryl rings stack on top of each other but to minimise interactions between the noncoplanar nitro-groups at C(13) and C(17) and the corresponding nitro-groups in neighbouring molecules, the rings are staggered with respect to their neighbours. The stacks of rings are parallel to the *a* axis, with the ring nuclei almost parallel to *c* (Figure 4). The indoline rings are packed parallel to each other up *c*, but are not overlapping as are the picryl rings.

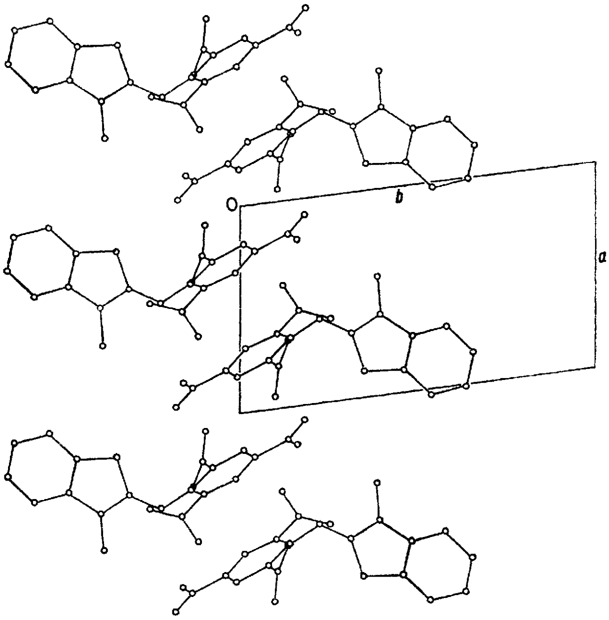

FIGURE 4 The crystal structure of 1-methyl-2-picryliminoindoline projected down *c*

The carbon–carbon bond lengths of the picryl nucleus are similar to those reported for 2,3,4,6-tetranitroaniline,[7] where the carbon–carbon bonds attached to the amine nitrogen are significantly larger than the normal (1·397 Å) reported for benzene,[15] and average 1·412 Å. The remaining four average 1·381 Å, which is significantly shorter than the benzene value. Also, in common with other polynitro-compounds,[7,13] the ring has internal angles significantly >120° at the carbon atoms attached to nitro-groups. This effect now appears to be general in such compounds.

The root-mean-square displacements along the principal axes of the thermal ellipsoids (Table 3) show definite evidence for considerable rigid-body motion of the indoline ring about N(11), since the magnitude of the principal displacement increases in the indoline ring with distance from this atom. The almost isotropic displacements in the picryl nucleus show that this unit remains essentially stationary during movement of the indoline ring. The nitro-groups show the expected rotational movement about the C–N vectors.

Conclusion.—Although the nature of these compounds is as expected (*i.e.* they are not oxidation–reduction

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[12] C. A. Coulson and A. Golebiewski, *Proc. Phys. Soc.*, 1961, **78**, 1310.

[13] A. S. Bailey and C. K. Prout, *J. Chem. Soc.*, 1965, 4867.

[14] J. T. Edsall, P. J. Flory, J. C. Kendew, A. M. Liquori, G. Nemethy, G. M. Ramachandran, and H. A. Scherago, *J. Mol. Biol.*, 1966, **15**, 399.

[15] A. Langseth and B. P. Stoicheff, *Canad. J. Phys.*, 1956, **34**, 350.

products), the crystal structure with the compound in the indoline form does not indicate whether one (or both) of the *o*-nitro-groups is locked in position by hydrogen bonding when in the indole form.

TABLE 3

The root-mean-square magnitudes and direction cosines of the principal axes of the thermal ellipsoids (the direction cosines are with respect to the crystallographic axes)

Atom	Axis i	U_i (Å)	l_i	m_i	n_i
O(1)	1	0·348	0·723	0·577	−0·460
	2	0·271	−0·662	0·815	0·212
	3	0·188	−0·199	−0·053	−0·862
O(2)	1	0·356	0·409	0·861	−0·187
	2	0·288	0·729	−0·414	0·376
	3	0·177	−0·549	0·297	0·908
O(3)	1	0·322	−0·102	0·957	0·231
	2	0·267	0·864	0·132	−0·705
	3	0·185	0·494	−0·260	0·671
O(4)	1	0·311	0·827	0·333	0·063
	2	0·244	−0·463	0·202	0·957
	3	0·188	−0·319	0·921	−0·284
O(5)	1	0·297	0·743	0·576	−0·360
	2	0·242	0·487	−0·497	0·604
	3	0·178	−0·460	0·649	0·711
O(6)	1	0·308	0·836	0·450	−0·325
	2	0·242	−0·047	0·133	0·945
	3	0·182	−0·547	0·883	−0·029
N(1)	1	0·254	0·250	−0·144	0·848
	2	0·230	−0·643	−0·686	0·326
	3	0·162	−0·724	0·714	0·419
C(2)	1	0·239	0·702	0·560	0·030
	2	0·196	0·134	−0·555	0·791
	3	0·171	−0·700	0·615	0·611
C(3)	1	0·244	0·104	−0·175	0·914
	2	0·219	0·723	0·604	−0·236
	3	0·178	−0·683	0·778	0·330
C(4)	1	0·270	0·327	−0·175	0·802
	2	0·236	0·939	0·052	−0·584
	3	0·221	0·106	−0·983	−0·126
C(5)	1	0·280	0·571	−0·274	0·599
	2	0·273	0·810	−0·104	−0·800
	3	0·212	−0·134	−0·156	−0·036
C(6)	1	0·289	0·970	0·115	−0·224
	2	0·271	−0·047	−0·182	0·962
	3	0·186	−0·238	0·977	0·158
C(7)	1	0·263	0·872	0·370	−0·171
	2	0·246	−0·043	−0·197	0·959
	3	0·186	−0·488	0·908	0·227
C(8)	1	0·239	0·763	0·247	0·269
	2	0·214	−0·403	−0·375	0·927
	3	0·185	−0·505	0·894	0·262
C(9)	1	0·238	0·712	0·271	0·334
	2	0·215	−0·390	−0·472	0·882
	3	0·189	−0·584	0·839	0·333
C(10)	1	0·338	0·241	−0·299	0·835
	2	0·262	−0·501	−0·795	0·104
	3	0·181	−0·831	0·528	0·541
N(11)	1	0·233	0·549	−0·008	0·623
	2	0·220	−0·452	−0·764	0·536
	3	0·170	−0·703	0·645	0·570
C(12)	1	0·204	0·192	−0·568	0·759
	2	0·189	0·498	0·708	0·171
	3	0·169	−0·846	0·420	0·628
C(13)	1	0·209	0·331	0·802	0·233
	2	0·187	−0·816	0·123	0·792
	3	0·180	0·475	−0·585	0·565
C(14)	1	0·214	0·206	−0·812	0·546
	2	0·205	0·598	0·495	0·314
	3	0·131	−0·774	0·311	0·776
C(15)	1	0·202	−0·034	−0·590	0·819
	2	0·189	0·659	0·512	0·199
	3	0·165	−0·752	0·624	0·538
C(16)	1	0·196	−0·150	−0·963	0·197
	2	0·193	0·328	−0·014	0·797
	3	0·181	0·933	−0·271	−0·571
C(17)	1	0·203	0·232	−0·928	0·336
	2	0·197	0·924	0·213	−0·101
	3	0·164	0·305	−0·306	−0·937
N(18)	1	0·263	0·974	0·077	−0·187
	2	0·208	−0·132	0·935	−0·370
	3	0·177	0·184	−0·345	−0·910
N(19)	1	0·227	−0·129	−0·463	0·902
	2	0·208	0·703	0·490	0·143
	3	0·178	−0·699	0·739	0·407
N(20)	1	0·276	0·531	0·704	0·117
	2	0·193	0·279	−0·584	0·705
	3	0·177	−0·800	0·404	0·699

EXPERIMENTAL

N.m.r. spectra were measured with a Perkin-Elmer R14 (100MHz) instrument and mass spectra with an AEI MS 9 instrument.

1-*Methyl-2-picrylaminoindole* (I; R^1 = Me, R^2 = H, R^3 = picryl).—1-Methylindole (2 g.; distilled from sodium) [16] and picryl azide (3 g.) were dissolved in ethyl acetate (30 ml.) and the resulting red solution was kept for 6 days. Nitrogen was evolved and solid started to separate after 6 hr. The solid was collected, washed with ethyl acetate, and dried (3·28 g., 78%). (With dimethyl sulphoxide as solvent the reaction appeared to be complete in 4 hr.). Crystallisation from dioxan gave material which softened and slowly decomposed without melting above 170°. From dimethylformamide the *compound* crystallised with solvent (n.m.r.); it softened at 180° and decomposed at 210° (Found: C, 50·4; H, 3·5; N, 18·8, 19·2 $C_{15}H_{11}N_5O_6$ requires C, 50·4; H, 3·1; N, 19·5%). N.m.r. spectra: τ ($CF_3{\cdot}CO_2H$) 0·95 (1H, d, *J* 2 Hz), 1·08 (1H, d, *J* 2 Hz), 1·8—2·5 (5H, m), and 5·67 (3H, s, NMe); τ [$(CD_3)_2SO$] −0·2 (NH), 1·3 (1H, d, *J* 2 Hz), 1·82 (1H, d, *J* 2 Hz), 1·8—2·6 (5H, m), and 6·4 (3H, s, NMe).

1,3-*Dimethyl-2-picrylaminoindole* (I: $R^1 = R^2$ = Me, R^3 = picryl) was prepared from 1,3-dimethylindole (4 g.; obtained by methylation of skatole [16]) and picryl azide (6 g.) in ethyl acetate (45 ml.) (yield 67% after 6 days). The *compound* formed yellow needles from n-propanol, darkening at 229°, m.p. 240—241° (decomp.) (Found: C, 51·5; H, 3·5; N, 19·0. $C_{16}H_{13}N_5O_6$ requires C, 51·8; H, 3·5; N, 18·9%), τ ($CF_3{\cdot}CO_2H$) 0·80 (1H, d, *J* 2 Hz), 1·24 (1H, d, *J* 2Hzh, 1·58 (1H, split d, *J* 7 and 2 Hz), 1·8—2·4 (3H, m), 6·01 (3H, s, NMe), and 7·12 (3H, s, CMe); τ [$(CD_3)_2SO$] −1·75 (NH), 1·30 (1H, d, *J* 2 Hz), 1·92 (1H, d *J* 2 Hz), 1·9—2·5 (4H, m), 6·38 (3H, s, NMe), and 7·5 (3H, s, CMe).

The reaction between indole-*N*-acetic acid (2·1 g.) and picryl azide (3·0 g.) in ethyl acetate (70 ml.) was slow. After 11 days the solid (36% yield) was collected. Recrystallisation from acetic acid gave the *compound* (I; R^1 = $CH_2{\cdot}CO_2H$, R^2 = H, R^3 = picryl) as yellow needles which contained acetic acid (n.m.r., and i.r. band at 1705 cm.$^{-1}$) and which darkened at 210° and decomposed between 254 and 290° (Found: C, 47·8; H, 3·0; N, 16·8. $(C_{16}H_{11}N_5O_8)_2$, $C_2H_4O_2$ requires C, 47·4; H, 3·0; N, 16·2%), τ [$(CD_3)_2SO$] −0·23 (NH), 1·27 (1H, d, *J* 2 Hz), 1·79 (1H, d, *J* 2 Hz), 1·9—2·8 (5H, m), 5·12 (2H, s, $N{\cdot}CH_2{\cdot}CO_2H$), and 8·09 (acetic acid of crystallisation); τ (D_2O–Na_2CO_3) 1·26 (1H, d, *J* 2·5 Hz), 1·68 (1H, d, *J* 2·5 Hz), 2·0—2·8 (5H, m), and 5·03 (2H, s). The compound decomposed in the mass spectrometer inlet (270°).

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[16] K. T. Potts and J. E. Saxton, *Org. Synth.*, 1960, **40**, 68.

TABLE 4

Observed structure amplitudes and calculated structure factors

l	|Fo|	Fc
0	0	
1	1755	-1733
2	3186	-3136
3	578	[illegible]
4	[illegible]	[illegible]
6	[illegible]	[illegible]
9	[illegible]	[illegible]
0	1	
-9	303	323
-5	257	-253
-4	2172	-2073
-3	623	-606
-2	[illegible]	[illegible]
-1	[illegible]	-1353
[illegible]	[illegible]	[illegible]
2	[illegible]	793
3	[illegible]	[illegible]
3	316	351
5	[illegible]	164
8	210	[illegible]
0	[illegible]	
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	-272
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
1	[illegible]	377
[illegible]	[illegible]	1188
[illegible]	[illegible]	1176
4	[illegible]	[illegible]
5	[illegible]	[illegible]
5	[illegible]	[illegible]
7	[illegible]	[illegible]
[illegible]	[illegible]	133
0	3	
[illegible]	360	[illegible]
-7	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	1108	[illegible]
-4	[illegible]	[illegible]
-3	[illegible]	[illegible]
-2	316	-297
-1	1225	[illegible]
0	1714	[illegible]
[illegible]	791	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	535
[illegible]	261	[illegible]
5	539	614
[illegible]	[illegible]	[illegible]
8	[illegible]	-623
0	4	
-8	372	397
-7	658	-675
-5	117	93
-4	798	763
-3	232	224
-2	679	-717
-1	[illegible]	[illegible]
0	[illegible]	-3168
1	[illegible]	[illegible]
2	[illegible]	[illegible]
3	[illegible]	[illegible]
4	[illegible]	324
5	[illegible]	145
8	[illegible]	-215
0	5	
-8	[illegible]	[illegible]
-7	[illegible]	[illegible]
-5	[illegible]	430
[illegible]	[illegible]	200
-4	[illegible]	[illegible]
-3	[illegible]	[illegible]
-2	[illegible]	[illegible]
-1	[illegible]	[illegible]
0	[illegible]	-457
1	[illegible]	[illegible]
2	[illegible]	[illegible]
[illegible]	[illegible]	-103
0	6	
-7	[illegible]	[illegible]
-4	[illegible]	[illegible]
[illegible]	192	75
[illegible]	[illegible]	-749
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
2	[illegible]	[illegible]
3	567	[illegible]
4	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
7	227	[illegible]
0	[illegible]	
[illegible]	379	[illegible]
[illegible]	[illegible]	[illegible]
-3	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
6	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
0	[illegible]	
[illegible]	[illegible]	[illegible]
-2	[illegible]	[illegible]
-1	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
3	392	[illegible]
[illegible]	[illegible]	[illegible]
5	[illegible]	[illegible]
6	[illegible]	[illegible]
0	[illegible]	
-6	[illegible]	[illegible]
-5	[illegible]	[illegible]
-4	578	[illegible]
-3	222	[illegible]
-2	609	[illegible]
-1	416	[illegible]
0	460	[illegible]
3	657	[illegible]
4	1164	[illegible]
5	698	[illegible]
7	146	[illegible]

l	|Fo|	Fc
0	10	
-9	153	160
-6	210	217
-5	429	-432
-4	406	410
-3	275	256
-1	112	-150
1	1079	1098
3	801	-810
6	324	310
0	11	
-6	192	197
-5	434	-429
-4	345	361
-3	390	401
-2	661	-702
-1	137	-131
0	200	152
1	523	523
6	164	137
0	12	
-3	152	148
-2	831	-853
-1	748	800
1	609	-605
2	144	135
6	154	134
0	13	
-3	679	672
-2	669	-664
0	147	129
1	327	-326
3	154	157
4	282	262
0	14	
-5	224	205
-4	179	-185
0	138	-155
2	195	175
4	180	-183
0	15	
-3	139	127
2	192	-206
0	16	
-2	147	126
0	156	-149
1	-16	
-1	195	-155
0	150	-120
2	210	185
1	-15	
-3	226	-186
-2	342	-310
-1	248	227
4	229	-227
1	-14	
-3	122	95
-2	319	-312
-1	159	149
0	104	-93
2	256	-231
3	210	217
4	131	126
1	-13	
-5	175	152
-2	210	-243
-1	257	263
0	428	-419
2	430	421
3	149	-170
6	150	-137
1	-12	
-5	203	-197
-3	217	230
-2	181	-139
-1	180	155
1	484	-513
2	695	725
3	226	-217
4	230	-218
1	-11	
-3	525	543
-2	344	347
-1	688	-693
0	316	293
1	191	161
2	151	170
3	470	-505
6	333	325
1	-10	
-6	224	-208
-5	285	273
-4	214	213
-3	360	-357
-2	224	232
-1	698	671
0	165	-146
1	199	-200
3	212	-200
4	460	477
1	-9	
-4	123	105
-3	382	403
-2	435	-423
-1	723	756
0	244	262
1	467	-496
2	227	-264
3	344	364
6	297	332
7	167	161
9	174	140

l	|Fo|	Fc
1	-8	
-8	169	141
-7	334	-320
-6	119	105
-5	581	500
-4	583	-555
-3	207	195
-2	512	562
-1	95	-73
0	503	466
1	237	-252
2	962	-889
3	396	399
4	219	251
7	169	157
8	159	134
1	-7	
-9	151	143
-7	165	179
-6	326	-314
-4	194	184
-2	750	684
-1	1365	-1237
2	535	486
4	226	-214
6	177	-198
8	191	160
1	-6	
-9	215	-210
-6	165	163
-5	251	-218
-3	307	-315
-2	424	-373
-1	1953	-1822
0	2321	2270
1	417	427
2	126	124
3	642	-555
4	145	181
5	166	-169
6	320	-350
7	151	-178
8	292	317
1	-5	
-7	158	-215
-6	414	420
-5	234	-203
-4	1427	-1428
-3	520	-556
-2	1577	1475
-1	1181	-1105
0	1362	-1345
1	290	374
2	2085	2012
3	775	-680
5	309	-283
7	513	-548
8	170	-146
1	-4	
-8	142	163
-7	268	-290
-5	180	-200
-4	251	-287
-3	269	-275
-2	1934	-1957
-1	457	-461
0	953	-950
1	752	742
2	2013	1968
3	831	-723
4	212	192
5	639	-554
6	655	-608
7	267	315
9	148	-167
1	-3	
-8	166	148
-7	410	-449
-6	368	419
-4	761	-770
-3	683	-656
-2	1383	-1403
-1	335	301
0	175	-127
1	240	192
2	727	694
3	2058	-2017
4	531	-526
5	1426	1442
6	604	-597
1	-2	
-9	186	155
-8	511	567
-7	452	-534
-6	280	-274
-5	339	368
-4	1270	1144
-3	1396	-1360
-2	132	-136
-1	1797	1877
0	670	623
1	4742	-4832
2	2526	-2471
3	2131	2138
4	354	352
5	130	108
6	567	-563
7	444	427
9	151	-139
1	-1	
-6	330	324
-5	149	118
-4	1540	1478
-3	286	-298
-2	2592	2671
-1	1395	-1380
0	1987	-1892
1	744	696
2	712	-711
3	966	-919
5	252	263
7	441	443
9	151	-131

l	|Fo|	Fc
1	0	
-7	359	379
-6	192	216
-5	355	-350
-4	1494	1403
-3	668	715
-2	2273	2275
-1	2633	2765
0	505	-539
1	1055	-1031
2	277	271
3	1334	-1273
5	552	586
6	527	545
1	1	
-7	305	305
-5	672	-601
-4	400	403
-3	4077	4207
-2	1437	1485
-1	2234	-2230
0	744	802
1	1101	1071
2	509	465
4	182	-147
5	229	252
6	432	445
7	238	-238
9	231	214
1	2	
-7	251	-227
-6	280	-258
-5	1035	1000
-3	585	-536
-2	214	-175
-1	106	97
0	1775	1719
1	2928	2863
3	903	826
4	221	-198
6	152	-141
1	3	
-6	447	453
-5	555	-515
-4	575	-607
-3	1625	-1646
-2	496	-560
-1	1871	1945
0	670	645
1	479	518
2	542	542
3	1941	1950
4	714	-682
6	190	-212
1	4	
-9	166	183
-8	169	180
-7	698	-700
-6	789	774
-5	624	-675
-4	692	-679
-3	673	-698
-2	1479	-1491
-1	845	-840
0	871	855
1	401	366
2	250	-171
3	1482	1483
5	395	-390
6	317	-358
7	268	302
1	5	
-8	334	317
-7	360	-352
-6	428	453
-5	254	-245
-4	711	-699
-3	294	-317
-2	1660	-1638
-1	1157	-1129
0	90	-50
1	343	347
2	303	259
3	532	-553
4	321	334
6	577	-604
7	275	284
1	6	
-6	164	151
-4	683	-638
-3	518	466
-2	1269	-1199
0	231	201
1	1401	-1371
2	250	-265
3	442	-457
4	140	-81
5	163	200
7	138	135
8	161	-137
1	7	
-8	191	-172
-4	648	-632
-3	1499	1452
-2	510	509
-1	857	-833
0	1150	-1123
1	122	47
2	862	-851
3	303	-287
4	269	249
5	139	134
6	336	-350
1	8	
-8	188	-157
-5	192	201
-4	126	143
-3	518	539
-2	612	571
-1	223	209
0	1714	-1629
1	964	946
2	131	123
3	509	-524
4	669	-655
6	375	373

l	|Fo|	Fc
1	9	
-5	189	-194
-4	559	560
-3	253	243
-2	135	-108
-1	731	732
0	272	277
1	126	96
2	1019	-1045
3	141	-146
4	619	612
1	10	
-7	309	-293
-5	201	220
-3	179	-190
-2	548	568
-1	328	359
0	514	-550
2	670	631
5	123	-85
1	11	
-5	161	190
-4	140	115
-3	393	-403
-2	474	-482
-1	354	353
0	882	864
3	150	127
5	135	-82
1	12	
-7	255	248
-6	153	148
-5	183	-164
-4	639	-630
-3	208	209
-2	390	407
0	276	-254
2	123	119
1	13	
-6	241	-233
-4	197	21
-3	247	262
-2	729	-726
1	241	242
2	256	202
1	14	
-4	367	-344
-3	210	195
-2	354	-351
0	179	199
1	166	-169
1	15	
-1	131	-119
1	319	-292
1	16	
-1	138	-139
2	-16	
0	138	156
2	-14	
-5	185	-189
-1	232	-212
0	107	-121
1	148	153
4	483	-481
5	171	157
2	-13	
-3	144	-124
-2	257	-261
-1	248	-258
0	331	359
1	233	228
2	430	-440
3	160	-151
4	132	112
2	-12	
-6	175	161
-3	155	-137
-1	287	289
0	460	-476
1	462	-463
2	597	626
3	358	-362
4	150	158
2	-11	
-5	290	281
-4	166	215
-3	225	205
-2	582	-654
-1	530	547
0	286	311
2	981	969
7	151	-164
2	-10	
-7	172	117
-5	276	292
-4	313	-358
-3	490	478
-2	329	321
-1	180	-119
1	740	-712
2	874	848
3	248	243
2	-9	
-8	151	-84
-7	253	279
-5	532	-532
-4	1060	1099
-3	257	-262
-2	237	-218
-1	194	194
0	271	274
1	237	206
2	599	604
3	150	-143
4	229	-294
5	747	800
6	170	-181

l	|Fo|	Fc
2	-8	
-9	358	-311
-7	227	227
-6	281	283
-5	1139	-1164
-4	1129	1142
-3	355	-365
-2	435	-463
-1	569	556
0	950	932
1	385	-337
2	398	-403
3	952	926
5	309	336
6	126	-125
2	-7	
-6	345	356
-5	1149	-1158
-4	505	513
-3	271	-297
-2	497	494
-1	592	-598
0	359	-286
1	1292	1292
3	411	-407
4	138	-166
6	202	210
7	179	187
2	-6	
-7	157	147
-6	158	175
-5	733	-779
-4	335	335
-3	913	-887
-2	473	-367
-1	1554	1504
0	132	-149
1	569	-495
3	971	-955
5	237	266
7	224	197
2	-5	
-8	167	-166
-6	139	173
-5	485	-485
-4	564	-579
-3	934	889
-2	514	-547
-1	256	335
1	1003	-992
2	449	-426
3	428	-429
4	263	-274
5	247	-236
8	231	268
2	-4	
-7	364	-377
-6	131	-163
-5	1085	1070
-4	663	-628
-3	904	-860
-2	92	-151
-1	1522	1500
0	750	-738
1	899	-914
2	1021	-972
3	365	342
4	481	-465
5	601	-593
7	165	135
2	-3	
-9	312	-300
-7	181	167
-5	208	-190
-1	1122	1076
0	1919	-1903
1	955	-909
2	303	296
3	185	186
4	239	-269
6	619	-618
8	221	-207
2	-2	
-9	155	-135
-7	126	-122
-5	564	-541
-4	960	557
-3	937	900
-2	1285	-1202
-1	2729	-2781
0	461	458
2	1411	1391
3	157	-203
4	270	-269
5	935	941
6	889	-897
7	156	-163
8	248	-229
2	-1	
-9	160	-167
-8	262	254
-4	983	977
-3	720	-650
-2	422	-436
-1	2458	-2444
0	768	774
1	618	625
2	965	928
4	117	101
5	1147	1155
6	901	-918
7	295	-271
2	0	
-7	273	244
-5	245	275
-4	1296	1251
-3	1721	-1677
-2	537	-458
-1	1345	1299
0	1074	-1020
1	248	252
2	444	401
3	378	412
4	226	255
5	600	618
6	202	-205
7	117	-100
8	193	175

l	|Fo|	Fc
2	1	
-9	182	191
-8	166	-196
-6	743	730
-5	131	93
-4	272	266
-3	980	-906
-2	127	139
-1	3378	4339
0	3944	-4055
1	791	791
2	645	658
3	293	250
8	378	354
2	2	
-8	151	153
-7	125	151
-6	234	228
-5	354	-333
-4	96	29
-3	783	800
-2	632	-665
-1	4627	5088
0	3393	-3471
1	1086	1024
2	931	908
3	552	-550
4	571	-546
5	159	-135
6	537	536
7	249	254
8	185	157
2	3	
-9	175	164
-6	615	-603
-4	275	293
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1	761	-704
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3	466	-458
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-1	153	131
0	813	-820
1	269	249
3	169	-173
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2	9	
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-2	230	179
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1	571	-600
3	300	304
4	313	318
5	244	-246

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3	906	-895
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-3	166	-212
-2	158	-214
-1	650	745
0	317	327
1	165	-201
2	126	168
3	820	-815
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2	12	
-6	215	-236
-4	159	-159
-3	236	242
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1	147	-149
2	306	303
3	346	-328
4	173	153
2	13	
-6	264	-243
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-2	354	362
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2	138	104
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2	15	
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3	259	-261
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-2	272	310
1	288	-324
2	231	227
5	168	-172
3	-12	
-4	142	-186
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-1	775	-852
1	415	417
3	220	-235
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3	-11	
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-2	217	-208
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2	173	-179
6	221	-207
3	-10	
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-1	206	213
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1	275	294
2	621	-685
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5	148	-149
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-8	196	201
-7	175	-191
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-4	121	132
-3	238	251
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2	407	-403
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-5	197	-206
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-3	487	505
-2	457	441
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2	329	361
4	137	-181
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7	169	109

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-6	257	[illegible]
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-6	217	265
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[illegible]	[illegible]	[illegible]
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0	[illegible]	[illegible]
1	[illegible]	[illegible]
2	[illegible]	[illegible]
3	[illegible]	[illegible]
4	[illegible]	[illegible]

TABLE 4 (*Continued*)

l	$\lvert F_o\rvert$	F_c
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-3	140	128
-2	595	-559
-1	395	367
0	269	-245
1	336	274
2	132	-163
3	227	276
4	471	463
5	632	653
3	3	
-8	117	-123
-6	245	314
-4	600	570
-2	481	-461
-1	844	799
0	501	-408
1	1042	-954
2	107	-126
3	467	462
4	610	604
5	334	345
3	4	
-5	175	-146
-4	311	335
-3	656	627
-2	306	-290
-1	987	-953
0	287	-270
1	211	162
2	706	678
3	625	-648
4	235	270
3	5	
-8	217	-243
-7	412	408
-6	269	-284
-5	339	299
-3	980	-830
-2	291	-268
-1	783	704
0	412	417
1	585	-547
2	496	469
3	639	-611
5	201	-213
6	164	144
3	6	
-8	446	-418
-7	691	679
-6	138	-175
-5	394	-386
-4	505	-505
-3	162	-193
-2	573	529
-1	156	195
0	223	-224
1	209	-178
2	498	516
3	195	-228
4	368	-357
5	230	-222
7	196	-205
3	7	
-8	162	-181
-7	317	321
-6	260	-277
-5	146	-185
-4	177	144
-3	774	-764
-2	96	88
0	96	65
1	291	259
3	196	208
4	389	-396
5	547	-541
6	275	285
3	8	
-7	339	341
-6	153	-150
-5	605	-598
-4	503	483
-3	427	-439
-2	128	-142
0	142	148
1	430	433
2	309	-345
3	219	-216
4	198	197
5	169	-152
3	9	
-7	165	-168
-4	537	541
-3	199	-234
-2	358	-374
0	297	296
1	506	-551
3	208	205
3	10	
-7	130	-96
-6	178	160
-5	172	177
-3	136	135
-1	530	-558
0	437	459
1	240	-266
3	222	209
3	11	
-5	146	163
-4	123	-93
-2	435	425
-1	219	-244
0	133	-156
3	12	
-3	226	250
0	116	-114
3	14	
-3	300	-275
-2	199	190
3	15	
-3	172	-202
4	-16	
0	256	-237
4	-15	
0	354	-351
1	135	122
4	-14	
1	207	216
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-1	284	290
0	108	92
1	243	-244
3	258	-253
4	-12	
-6	234	-226
-1	233	244
0	310	289
1	459	-463
2	151	-135
4	228	205
5	209	-218
4	-11	
-4	405	-410
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-1	155	-157
0	223	-221
1	373	390
3	176	-208
4	-10	
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-5	165	-145
-3	284	-295
0	265	-303
1	621	643
2	613	-651
5	159	151
6	166	-153
4	-9	
-8	138	108
-7	158	152
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-5	202	-197
-4	165	180
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-1	548	-567
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2	352	-346
3	298	310
4	-8	
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1	1016	1023
2	163	-153
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4	-7	
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-1	247	287
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2	707	720
3	206	-232
4	423	460
5	125	-144
4	-6	
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-8	153	148
-7	295	-276
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-2	469	-453
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2	249	240
4	428	453
5	241	-262
4	-5	
-9	210	-205
-8	175	-211
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-4	976	-952
-3	559	-514
-2	675	594
-1	691	720
0	713	687
2	201	-210
4	-4	
-6	1024	-1026
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-4	295	-282
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-1	430	409
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1	390	-355
2	546	-551
3	439	425
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1	980	984
2	100	77
3	908	-964
4	359	-395
5	376	409
6	157	120
4	-2	
-8	369	395
-6	173	-202
-5	572	-587
-4	146	150
-3	600	-534
-2	447	-381
-1	274	-341
0	90	93
1	928	-883
2	514	-494
3	444	-436
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7	138	-115
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-5	183	177
-3	334	265
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0	493	-473
2	854	-854
3	179	-165
4	469	494
5	679	-683
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4	0	
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2	253	242
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4	357	369
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1	1291	1254
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6	150	-161
4	3	
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6	172	183
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5	188	-151
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0	152	149
1	214	224
2	253	-254
3	188	-188
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1	330	-351
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5	294	-280
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-4	304	-316
-3	242	223
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2	526	528
3	344	-353
4	182	-228
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0	954	936
1	694	-711
2	172	174
3	135	144
5	166	-149
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-5	214	-211
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4	198	226
6	238	242
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-2	196	-205
-1	696	-701
1	363	353
2	304	-287
3	285	-302
4	332	349
5	386	379
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-7	433	-451
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-1	825	-820
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5	232	249
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-9	373	-382
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3	276	267
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-5	214	-258
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1	653	-593
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3	161	-166
4	231	-250
5	-1	
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-6	196	-195
-5	454	-424
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-3	302	-270
-2	342	-391
-1	139	152
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-6	205	-221
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4	344	-347
6	132	-129
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-6	146	-162
-5	158	-153
-4	193	203
-3	346	-344
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1	219	-188
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3	227	-223
4	342	-342
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6	210	-183
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-8	155	112
-7	134	105
-6	393	381
-5	485	-464
-4	460	465
-3	486	516
-2	131	162
0	204	172
1	134	-133
2	123	-112
3	296	273
4	275	-307
5	3	
-6	318	324
-5	219	222
-4	432	431
-2	440	442
-1	325	-332
0	102	144
1	453	440
2	201	-212
4	281	276
5	4	
-8	248	-250
-6	160	168
-5	142	152
-4	213	180
-3	219	-197
-2	601	607
-1	267	304
0	364	-380
1	285	-260
2	423	413
4	125	147
5	5	
-8	154	-151
-7	272	-263
-5	227	275
-4	177	182
-3	119	113
-1	383	-398
0	240	267
1	152	-180
2	149	-138
3	650	663
5	135	-122
5	6	
-9	185	-197
-7	143	-142
-5	215	237
-4	439	-465
-3	242	-271
-2	633	647
-1	460	-496
0	487	-449
1	695	735
2	299	-286
5	7	
-8	408	421
-6	286	-272
-5	232	-223
-4	248	285
-2	281	-250
-1	203	231
0	232	-233
2	190	-188
5	132	127
5	8	
-8	314	280
-7	369	-360
-6	155	157
-4	334	-328
-3	418	472
-2	226	-239
-1	321	-376
0	163	171
1	265	274
2	235	-196
5	9	
-7	257	-293
-6	139	-160
-5	494	523
-4	472	-468
-3	249	-269
-2	188	167
-1	234	293
3	150	-122
4	148	-114
5	10	
-4	211	-216
-2	119	75
-1	136	95
1	142	-92
2	128	123
5	11	
-5	145	146
-4	133	-116
5	12	
-1	212	196
0	157	-167
5	13	
-3	125	68
-2	193	-186
6	-14	
-3	157	-172
-1	194	-175
0	385	379
6	-13	
1	151	-134
6	-12	
-2	187	-177
-1	126	-127
3	170	161
6	-11	
-6	206	189
-5	135	-91
-4	246	-280
-3	141	163
-2	157	175
-1	191	-192
0	200	-213
1	257	256
2	345	-343
3	141	-153
6	-10	
-4	150	156
-2	152	158
-1	308	307
0	437	-434
1	390	-419
3	214	230
4	350	-344
6	-9	
-7	147	-140
-6	184	199
-5	143	-126
-3	523	551
-2	213	-217
-1	322	-312
0	691	677
1	166	-193
2	393	-379
6	-8	
-7	186	-208
-5	271	250
-4	398	-388
-2	564	580
0	321	320
1	586	-605
2	354	395
3	206	-201
6	-7	
-7	171	169
-6	444	-447
-5	292	-293
-4	437	430
-3	694	706
-2	787	-784
-1	463	-464
0	1488	1499
1	669	-711
2	273	257
3	229	237
6	-6	
-8	227	-230
-7	131	-123
-6	502	494
-4	641	-649
-1	827	-830
0	1064	1069
2	160	-135
3	487	511
6	-5	
-6	406	411
-5	339	-376
-3	748	-779
-2	131	120
-1	149	161
0	216	-228
1	290	305
3	455	475
6	-4	
-9	171	156
-8	288	-260
-6	1313	1360
-5	1072	-1107
-3	317	313
-2	369	-383
0	336	-342
1	187	162
2	286	268
3	256	265
4	271	-280
6	-3	
-8	294	318
-7	550	-544
-6	728	752
-5	133	-118
-4	260	-258
-3	380	345
-1	434	-445
1	158	173
6	-2	
-9	183	-176
-8	130	142
-7	153	-131
-6	352	-375
-5	224	203
-4	411	407
-2	179	-158
-1	273	289
0	134	138
1	336	-330
2	320	-339
3	420	-425
4	130	140
6	-1	
-9	133	137
-8	286	-275
-7	237	-237
-6	323	-315
-4	253	263
-3	466	473
-2	202	-210
-1	227	224
0	186	205
1	746	-754
2	312	-302
6	0	
-8	154	-114
-7	315	-324
-6	166	-133
-5	145	146
-4	264	-272
-3	309	316
-2	135	168
-1	200	-228
0	160	76
2	233	-217
4	245	-261
5	238	244
6	1	
-7	[illegible]	-175
-6	158	-195
-5	209	-230
-3	108	-85
-2	131	113
-1	295	304
0	99	78
1	591	-603
3	442	427
4	239	-236
6	2	
-6	303	313
-5	489	[illegible]
-3	361	394
-1	745	-763
1	180	161
2	332	360
3	234	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	357
6	3	
-8	[illegible]	153
[illegible]	[illegible]	[illegible]
-3	731	[illegible]
-2	344	-361
-1	541	[illegible]
0	340	[illegible]
1	292	-292
3	163	[illegible]
4	[illegible]	[illegible]
6	4	
-7	363	363
[illegible]	323	306
[illegible]	696	[illegible]
-3	615	[illegible]
-2	533	-514
1	327	320
6	5	
-7	226	-249
-6	152	177
-5	170	165
-4	255	239
-3	182	216
-2	1114	[illegible]
-1	211	[illegible]
2	277	260
6	6	
-7	143	153
-6	142	136
-5	192	-206
-4	194	-205
-3	652	693
-2	752	-768
-1	322	353
0	374	395
1	265	-234
6	7	
-8	236	-251
-5	177	168
-4	217	-202
-3	211	205
1	212	-251
6	8	
-7	269	240
-5	166	-182
-4	145	140
-1	171	-176
6	9	
-5	169	-150
6	11	
-2	159	-170
7	-13	
-3	163	167
-1	326	-290
0	138	108
7	-12	
-4	196	-189
7	-11	
0	182	-179
7	-10	
-6	248	213
-3	241	-258
0	283	-281
2	229	236
7	-9	
-7	146	-146
-6	159	110
-4	243	252
-3	364	-367
1	359	-362
3	126	98
7	-8	
-4	304	279
-3	142	-167
-2	242	248
-1	215	-205
2	172	-227
7	-7	
-7	194	191
-4	122	120
-2	417	412
-1	345	343
0	420	-425
1	242	220
7	-6	
-5	390	-376
-3	572	610
-2	123	-109
-1	237	226
0	143	162
1	229	246
4	312	-296
7	-5	
-7	212	-221
-4	342	-344
-3	264	277
-2	237	260
0	413	419
1	236	212
2	312	-322
7	-4	
-7	101	-224
-4	175	197
-3	368	-373
-2	127	101
-1	[illegible]	139
0	96	67
7	-3	
-8	138	-133
-7	331	-345
-6	523	554
-5	219	-193
-3	202	-243
-2	480	-535
-1	209	252
0	187	199
1	401	-406
3	342	349
7	-2	
-8	176	177
-7	231	-226
-6	546	560
-4	536	-555
-3	529	577
-2	311	-310
-1	405	-440
0	230	-268
1	127	85
4	275	290
7	-1	
-5	280	[illegible]
-3	360	[illegible]
-2	523	-579
-1	134	-96
0	540	[illegible]
2	352	297
7	0	
-8	212	-188
-7	229	220
-6	196	204
-3	540	581
-2	513	-520
-1	301	297
1	386	366
2	[illegible]	-377
3	196	-192
7	1	
-8	148	-116
-7	149	-162
-5	245	252
-4	273	-295
-2	587	687
-1	148	177
0	603	-615
1	195	199
7	2	
-6	276	-282
-4	419	425
-1	174	170
1	299	318
2	189	[illegible]
3	256	-250
7	3	
-8	144	-126
-7	166	-176
-5	113	68
-4	367	-371
-3	164	-180
-2	329	324
-1	462	467
1	130	-98
7	4	
-6	347	-368
-1	160	120
7	5	
-8	147	-107
-4	201	-202
7	6	
-7	160	187
-5	175	-146
-3	226	247
-2	232	-193
7	7	
-3	239	235
-2	352	-320
1	146	-143
2	196	198
7	8	
-2	282	-269
0	196	220
1	191	-198
7	9	
-2	127	57
7	10	
-5	150	-194
-4	191	175
8	-11	
-4	155	-152
-3	209	165
8	-10	
-5	182	117
-3	316	-278
-1	124	97
8	-9	
-5	319	-340
-4	179	161
-2	128	-107
2	209	220
8	-8	
1	133	-98
8	-7	
-7	146	138
-6	158	-132
-4	201	230
-1	152	136
0	418	-440
8	-6	
-4	432	458
-3	285	-311
-2	137	-161
-1	637	677
0	624	-629
1	143	166
8	-5	
-6	256	264
-1	747	746
0	727	-708
2	201	203
8	-4	
-7	133	135
-6	323	-318
-4	162	171
-3	214	216
-2	248	-270
-1	268	233
0	106	-110
1	210	212
2	264	238
3	166	-202
8	-3	
-7	420	434
-6	544	-544
-5	149	159
-3	146	-112
-1	157	210
0	206	203
1	115	-27
2	272	300
3	201	-193
8	-2	
-8	143	-124
-7	426	424
[illegible]	592	-629
0	397	370
[illegible]	-1	
[illegible]	204	-210
[illegible]	223	177
-4	166	-153
-3	370	-379
[illegible]	0	
[illegible]	141	116
0	122	-102
8	1	
-6	215	206
0	347	-341
8	2	
-4	157	172
0	229	-225
1	117	-95
3	137	-126
8	3	
-3	146	136
0	213	-219
8	4	
-6	262	-245
-4	189	146
0	175	-169
2	195	185
8	5	
-6	194	-205
8	6	
-6	141	-148
-5	174	159
-3	414	-445
-2	389	400
8	7	
-3	405	-435
-2	377	399
0	143	-114
8	8	
-4	190	189
-3	312	-291
9	-9	
-1	144	-112
9	-8	
-4	218	-237
0	178	-146
9	-7	
-4	194	-190
-3	199	202
9	-6	
-4	185	-185
0	131	151
9	-5	
-6	148	-174
-5	156	133
-3	130	-93
0	130	110
9	-4	
-4	175	178
-3	136	-87
-1	142	151
0	142	117
9	-3	
-2	180	157
-1	174	176
0	124	-137
1	132	-113
9	-2	
-7	204	216
-6	203	-193
-3	130	90
9	-1	
-7	269	281
-6	192	-182
-3	130	-117
-2	168	168
9	0	
-7	213	204
-6	194	-201
-4	294	288
-3	258	-258
-1	133	-126
0	107	45
9	1	
-7	128	102
-5	132	-128
-4	210	228
-3	349	-331
0	124	138
9	3	
-2	155	-143
0	247	240
9	4	
-4	179	-168
-3	130	132
-2	219	182
-1	189	-157
10	-5	
-3	142	149
-2	229	108
10	-4	
-2	385	[illegible]
10	-3	
-2	203	190
-1	355	-317
10	-2	
-2	158	158
-1	237	-229
10	-1	
-3	145	-174
10	0	
-2	152	-159
10	1	
-4	252	-256
-2	147	119

Crystallographic Data

Large crystals of the *N*-methylindole adduct were obtained by slow crystallisation from ethyl acetate. Suitable crystals for *X*-ray investigation were obtained by partially dissolving a large crystal with dioxan. Approximate cell parameters were obtained from Weissenberg and precession photographs and were refined by use of a Hilger and Watts four-circle PDP 8 instrument (Y 290—FA 128), before commencement of data collection.

Crystal Data.—$C_{15}H_{11}N_5O_6$, $M = 357{\cdot}3$, Triclinic $a = 8{\cdot}01 \pm 0{\cdot}01$, $b = 13{\cdot}20 \pm 0{\cdot}01$, $c = 7{\cdot}79 \pm 0{\cdot}01$, $\alpha = 92{\cdot}7 \pm 0{\cdot}2$, $\beta = 107{\cdot}5 \pm 0{\cdot}2$, $\gamma = 96{\cdot}5 \pm 0{\cdot}2$, $U = 780{\cdot}2$ Å^3, $D_m = 1{\cdot}515$ (by flotation), $Z = 2$, $D_c = 1{\cdot}520$. Space group $P\bar{1}$ (C_i^1, No. 2). Mo-K_α radiation, $\lambda = 0{\cdot}7107$ Å, μ(Mo-K_α) = 1·31 cm.$^{-1}$.

Data were collected from an approximately rod-shaped crystal (0·8 × 0·6 mm. diam.), mounted about the rod axis (*c*). An ω—2θ scan was used to collect 3376 reflections in the range θ = 0—27°. In the range θ = 0—20° balanced filters (zirconium oxide–yttrium carbonate) were used; at $\theta > 20°$ a single zirconium oxide filter was employed.

Each reflection was scanned over 0·8°, counting at intervals of 0·02° sec.$^{-1}$, with background counted for 10 sec. on both sides of the scan. A measurement was discarded if the backgrounds differed by more than 15 σ * of their total count. Of the 3376 reflections counted 2051 had sufficiently equal backgrounds and an intensity $> 3\ \sigma$.

Solution and Refinement of the Structure.—Approximate atomic co-ordinates were generated by a symbolic addition program, developed by Hodder and Prout [17] for the English Electric KDF 9 computer. The computer chooses the origin determining reflections, and then those for which symbols are assigned are chosen one by one as required to continue the phase determination. This programme used all triple products of probability over 0·942 given by the 737 reflections with $E > 1{\cdot}1$. To fix the origin of the unit cell $03\bar{6}$, $22\bar{1}$, and $11\bar{3}$ were given positive signs. The signs of $6\bar{4}\bar{6}$, 094, and $21\bar{1}$ were represented by the symbols *A*, *B*, and *C* respectively. 381 signs with a probability $> 0{\cdot}9975$ were determined. The probability was then lowered to 0·880, and a further 180 signs were determined.

Using these 561 phased *E* values, Fourier syntheses were computed for the most probable signs of *A*, *B*, and *C*. The correct solution, with *A* and *C* positive, and *B* negative was found to be the third most probable (ignoring the solution for all three positive). In the correct solution only three phases (all of low probability) were subsequently shown to be incorrect. In the synthesis using these phases 26 large peaks (corresponding to the positions of all the nonhydrogen atoms) were observed.

In the full-matrix least-squares refinement of the trial structure, the quantity minimised was $\Sigma w(|F_o| - |F_c|)^2$. Isotropic temperature factors were assumed for all atoms. Scattering curves used were those given in ref. 18. After four cycles of refinement parameter shifts were considerably less than their estimated standard deviations and *R* was 0·128. From a difference Fourier map all the hydrogen atom positions were apparent, as well as considerable anisotropic movement in the indole ring. The co-ordinates of all but the *N*-methyl hydrogen atoms were deduced from the molecular geometry.

All nonhydrogen atoms were then assigned anisotropic temperature factors and a further six cycles of least-squares refinement were computed with alternating matrix blocking until convergence was achieved; in the two final cycles two matrix blocks were used, one containing all space parameters and the other the F_c value, scale factor, and all temperature factors. Unit weights were used for $|F_o| < 2000$, otherwise the relation $\sqrt{\omega} = 2000/|F_o|$ was used.

TABLE 5

Fractional atomic co-ordinates ($\times 10^5$) with standard deviations in parentheses

Atom	*x*/*a*	*y*/*b*	*z*/*c*
O(1)	06535(42)	90023(24)	01858(36)
O(2)	32875(41)	86421(26)	06845(37)
O(3)	59838(35)	88152(24)	87691(36)
O(4)	40911(39)	74606(19)	76075(36)
O(5)	18881(35)	115887(20)	74822(34)
O(6)	07491(37)	118024(19)	46642(36)
N(1)	39844(37)	60669(21)	29688(40)
C(2)	32627(44)	68728(25)	34656(43)
C(3)	13524(45)	65057(25)	33253(49)
C(4)	11678(46)	53798(25)	27172(46)
C(5)	−02229(53)	46100(29)	23713(56)
C(6)	−00064(58)	36157(29)	18048(60)
C(7)	15994(59)	34265(28)	16101(58)
C(8)	30168(52)	41967(27)	19599(52)
C(9)	27705(47)	51673(25)	25201(47)
C(10)	58179(56)	61493(32)	29440(66)
N(11)	41584(36)	77631(20)	39855(37)
C(12)	34007(39)	85885(23)	44050(41)
C(13)	36876(40)	89630(24)	62165(41)
C(14)	31625(40)	98533(24)	67402(41)
C(15)	22145(39)	103950(23)	53834(41)
C(16)	18272(40)	100827(23)	35722(41)
C(17)	24546(38)	92018(23)	31276(37)
N(18)	46647(39)	83598(23)	76468(38)
N(19)	15798(34)	113374(20)	58843(38)
N(20)	21094(41)	89247(21)	11848(36)

TABLE 6

Anisotropic thermal parameters *

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
O(1)	0·08695	0·09222	0·03889	−0·01168	−0·00510	0·05620
O(2)	0·08997	0·11425	0·04973	0·01018	0·07159	0·06852
O(3)	0·05325	0·10128	0·04763	0·03875	0·00148	0·03190
O(4)	0·09010	0·04944	0·06206	0·03604	0·05730	0·06056
O(5)	0·07440	0·05978	0·04734	−0·01666	0·03827	0·05047
O(6)	0·07837	0·05235	0·05815	0·01421	0·03102	0·06792
N(1)	0·04840	0·04136	0·06191	−0·00107	0·05192	0·03207
C(2)	0·04931	0·04365	0·03813	0·00923	0·03727	0·03594
C(3)	0·04538	0·03961	0·05865	0·00108	0·04151	0·02381
C(4)	0·06151	0·04902	0·07067	0·00500	0·05313	0·01271
C(5)	0·07440	0·04557	0·07616	0·00210	0·04800	0·00234
C(6)	0·08256	0·03748	0·07245	−0·00019	0·04796	0·02921
C(7)	0·06489	0·04296	0·06005	0·00428	0·04199	0·03786
C(8)	0·05417	0·03902	0·04868	0·00889	0·04116	0·02645
C(9)	0·05293	0·04128	0·04945	0·00908	0·04172	0·02638
C(10)	0·06486	0·06242	0·10376	−0·01636	0·09893	0·03053
N(11)	0·04731	0·04099	0·04865	−0·00117	0·04554	0·02564
C(12)	0·03378	0·03628	0·03846	−0·00069	0·02952	0·01075
C(13)	0·03634	0·04090	0·03511	0·03196	0·02320	0·01878
C(14)	0·03550	0·04228	0·03460	−0·00420	0·02468	0·01878
C(15)	0·03339	0·03438	0·03786	−0·00198	0·02669	0·01389
C(16)	0·03471	0·03830	0·02686	0·00557	0·02518	0·01177
C(17)	0·03876	0·03913	0·02907	−0·00260	0·02373	0·00903
N(18)	0·06785	0·04235	0·03346	−0·00030	0·02990	0·02006
N(19)	0·04045	0·03922	0·04867	−0·00259	0·03124	0·01913
N(20)	0·05399	0·06100	0·04068	0·02608	0·04546	0·05490

* In the form: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{31}lhc^*a^* + 2U_{12}lka^*b^*)]$.

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* If the difference in backgrounds was $< 6\,\sigma$ they were averaged; from 6—15 σ the first background was used. This relatively loose limit was used because of very pronounced streaking induced by the crystal.

[17] O. J. R. Hodder and C. K. Prout, unpublished work.

[18] 'International Tables for *X*-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1962.

The final R was 0·053, at which point a difference Fourier map showed no peaks or holes $>0{\cdot}2$e.

In Table 4 are compared the observed structure amplitudes with structure factors calculated from the atom positional parameters (Table 5). Anisotropic temperature parameters are shown in Table 6.

Calculations were carried out on an English Electric KDF 9 computer. Isotropic refinement and Fourier synthesis were calculated using ' Novtape ', initially developed by J. S. Rollett *et al.*, with modifications by O. J. R. Hodder. Anisotropic refinement was programmed by G. C. Ford and J. S. Rollett. Calculation of anisotropic root-mean-square displacements was programmed by J. R. Carruthers.

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