Formation and Crystal Structure of 5-[Hydroxy(phenyl)amino]-3,3,5-trimethyl-2-phenylisoxazolidine and *N*-(3,3,5-Trimethyl-2-phenylisoxazolidin-5-yl)phenylaminyl *N*-Oxide (Banfield and Kenyon's Radical)

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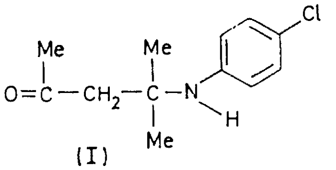
The product formed by the reaction of acetone with *N*-phenylhydroxylamine is (VI), 5-[hydroxy(phenyl)amino]-3,3,5-trimethyl-2-phenylisoxazolidine, and the corresponding stable free-radical (Banfield and Kenyon's radical) is (VII), *N*-(3,3,5-trimethyl-2-phenylisoxazolidin-5-yl)phenylaminyl *N*-oxide, and not *N*-(1,1-dimethyl-3-phenyliminobutyl)phenylaminyl *NN'*-dioxide or *N*-[1,1-dimethyl-2-(3-methyl-2-phenyloxaziridin-3-yl)ethyl]phenylaminyl *N*-oxide as was previously thought.

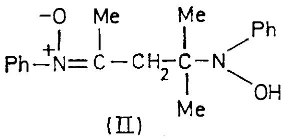
The crystal structure of two crystal forms [(A) and (B)] of (VI) has been determined by direct methods and refined by block-diagonal least-squares to R 0·093 (A) and 0·098 (B). In both forms the space group is $P2_1/n$, with $Z = 4$; for (A): a = 12·45(1), b = 13·25(1), c = 10·18(1) Å, β = 97·7°(1); and for (B): a = 13·96(1), b = 7·37(1), c = 16·14(1) Å, β = 94·1°(1). The molecular shape is the same in both types.

LARGE crystals are formed from concentrated solutions of *N*-phenylhydroxylamine in acetone which have been set aside at room temperature for a few days.[1] The reaction appears to involve the condensation (1).

$$2\mathrm{PhNHOH} + 2\mathrm{Me_2CO} \longrightarrow \mathrm{C_{18}H_{22}N_2O_2} + 2\mathrm{H_2O} \qquad (1)$$

Based on this molecular formula determined by Beckmann and Scheiber,[2] and the fact that the product could be hydrolysed by aqueous hydrochloric acid to 4-(4-chloroanilino)-4-methylpentan-2-one, (I), Banfield and Kenyon[3] suggested that the condensation product was either 3-[hydroxy(phenyl)amino]-1,3-dimethylbutylideneaniline *N*-oxide, (II) or 3-{2-[hydroxy(phenyl)-amino]-2-methylpropyl}-3-methyl-2-phenyloxaziridine, (III).


(I)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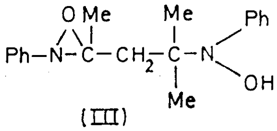
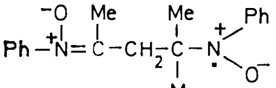

(III)

The particular interest in this condensation product is that, as discovered by Banfield and Kenyon, it is readily oxidised by ammoniacal silver nitrate to an extremely stable free-radical,[3,4] which has been widely used in e.s.r. work [5-16] and has always been assumed to be the nitroxide corresponding to (II) or (III), namely (IV) or (V).

We now have evidence from solution ^{1}H magnetic resonance spectroscopy and from an X-ray single-crystal structure determination that the condensation product has neither of these structures but is 5-[hydroxy(phenyl)-amino]-3,3,5-trimethyl-2-phenylisoxazolidine, (VI), and that the Banfield and Kenyon free-radical is the corresponding nitroxide (VII).[17]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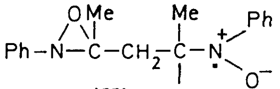
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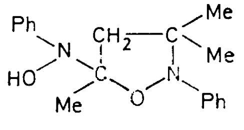
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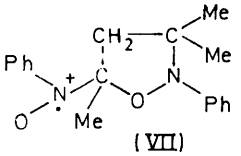
(VII)

RESULTS AND DISCUSSION

The ^{1}H n.m.r. spectrum of the condensation product has the following absorptions: δ ($\mathrm{CDCl_3}$) 7·83 (1H, s), 6·88—7·49 (10H, m), 3·14 (1H, d, J 13·0 Hz), 2·16 (1H, d, J 13·0 Hz), 1·41 (3H, s), 1·37 (3H, s), and 0·93 (3H, s). The AB system represented by the double doublet could be assigned to the methylene group in any of the structures (IV), (V), or (VI). If, however, the compound had the structure (II) these methylene protons would have to be non-equivalent because of restricted rotation within the molecule. No change was observed in the AB pattern as the temperature was raised to 60°. Also, no rotational restriction is observed either in (I) or in its phenylhydrazone. In each of these compounds, which may be taken as reasonable models

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for testing restricted rotation in (II), the methylene protons appear as a singlet.

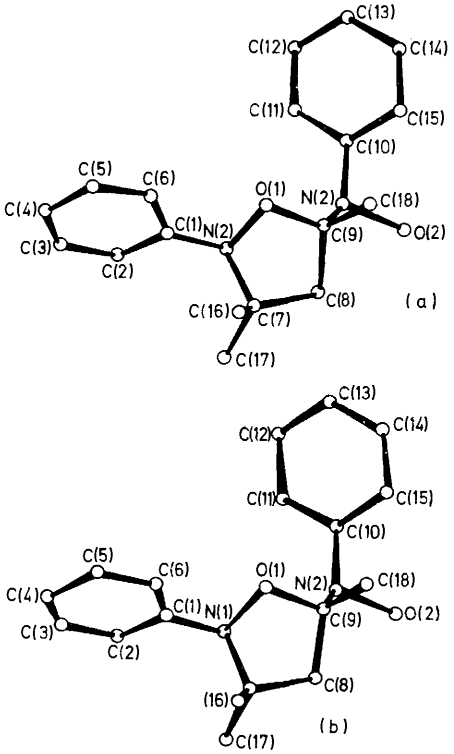

FIGURE 1 A perspective view of the molecule viewed along the normal to the mean plane of the five-membered ring: (a) type (A), (b) type (B)

The AB pattern could be accounted for by structure (III) since the methylenes are adjacent to an asymmetric carbon atom. Nevertheless, the coupling constant is remarkably large for such prochiral protons. Even more remarkable is the non-equivalence of the two *gem*-methyl groups. Although in principle this could be accounted for by the asymmetric centre in (III), the measurable size of this difference makes it unlikely. Admittedly the shift difference is small: indeed, in the measurements at 60 MHz the absorptions of these two methyl groups were initially recorded as a single singlet.[17,*] However, by replacing the chloroform solvent by benzene the chemical-shift difference is increased from 0·04 to 0·13 p.p.m. It is the solvent shift of the higher-field absorption which largely accounts for this increased difference [δ (C_6H_6) 1·38, 1·25]. This solvent change also gives rise to a solvent shift of the AB system [δ (C_6H_6) 3·03 (1H, d, 13·0 Hz) and 1·85 (1H, d, 13·0 Hz)]. The non-equivalence of the *gem*-methyl groups, the magnitude of the differences involved, and the size of J for the AB systems are best explained in terms of structure (VI) in which these atoms and groups are attached to ring carbon atoms. It is possible that of the *gem*-methyl groups, that which absorbs at higher field and is the more shifted by the solvent change is *cis* to the proton of the methylene group which has the higher-field absorption and is also the more shifted by the solvent change. Attempts to confirm this assignment from a nuclear Overhauser experiment using C_6D_6 as solvent were unsuccessful.

Unequivocal confirmation of structure (VI) as being the condensation product has been obtained from X-ray diffraction studies. Two crystal forms, both monoclinic, were examined and the results are shown in Figure 1, viewed along the normal to the mean plane of the five-membered ring (B). They are remarkably similar in conformation. Since a model of the molecule is quite flexible it might have been expected that it would have adopted a different shape under the different

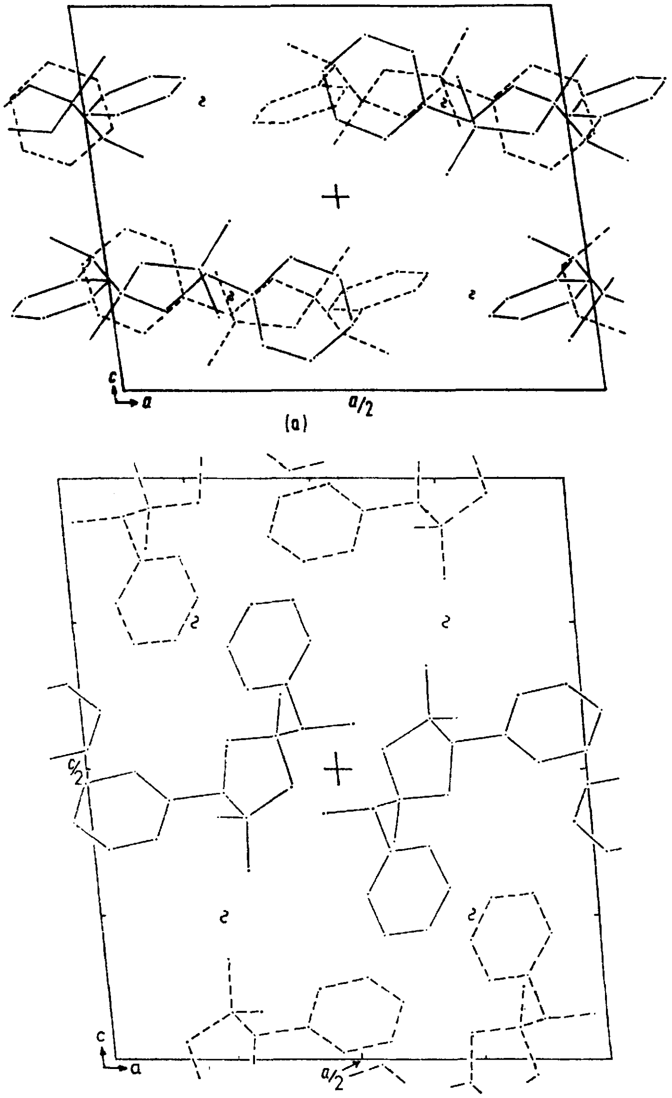

FIGURE 2 The packing of the molecules in the two unit cell: (a) type (A), (b) type (B). Broken lines refer to molecules which are at $b/2$

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[*] This is a reasonable general assignment for these methyl groups. An alternative possibility is that one of the *gem*-methyl groups is responsible for the absorption of δ ($CDCl_3$) 0·93 and an accidental coincidence of the other *gem*-methyl absorption with that of the 5-methyl group occurs. However, this seems unlikely.

[17] Preliminary report, R. Foster, J. Iball, and R. Nash, *Chem. Comm.*, 1968, 1414.

packing forces operating in the two crystal modifications. The methods of packing are indicated in Figure 2. Bond lengths and angles for the two forms are given in Table 1.

TABLE 1

Comparison of bond lengths (Å) and bond angles (deg.) in (VI) for both crystal types with estimated standard deviations in parentheses

(*a*) Distances

	(A)	(B)
C(1)–C(2)	1·407(17)	1·398(14)
C(1)–C(6)	1·382(16)	1·379(15)
C(1)–N(1)	1·425(14)	1·394(11)
C(2)–C(3)	1·384(18)	1·379(14)
C(3)–C(4)	1·350(22)	1·372(19)
C(4)–C(5)	1·380(20)	1·354(18)
C(5)–C(6)	1·379(18)	1·352(16)
C(7)–C(8)	1·514(17)	1·507(13)
C(7)–C(16)	1·514(17)	1·499(18)
C(7)–C(17)	1·525(18)	1·540(15)
C(7)–N(1)	1·507(13)	1·504(13)
C(8)–C(9)	1·519(16)	1·551(13)
C(9)–C(18)	1·543(17)	1·495(14)
C(9)–N(2)	1·505(13)	1·503(13)
C(9)–O(1)	1·411(13)	1·384(10)
C(10)–C(11)	1·376(16)	1·366(13)
C(10)–C(15)	1·398(16)	1·376(12)
C(10)–N(2)	1·449(14)	1·440(11)
C(11)–C(12)	1·388(18)	1·418(15)
C(12)–C(13)	1·377(20)	1·356(17)
C(13)–C(14)	1·398(20)	1·367(15)
C(14)–C(15)	1·370(19)	1·403(15)
N(1)–O(1)	1·460(11)	1·470(9)
N(2)–O(2)	1·448(11)	1·425(9)

(*b*) Angles

	(A)	(B)
C(2)C(1)C(6)	117·5(1·0)	121·5(0·9)
C(2)C(1)N(1)	116·5(1·0)	123·0(0·9)
C(6)C(1)N(1)	126·0(1·0)	115·5(0·9)
C(1)C(2)C(3)	119·4(1·2)	117·2(1·1)
C(2)C(3)C(4)	122·1(1·3)	120·6(1·1)
C(3)C(4)C(5)	119·4(1·2)	121·0(1·1)
C(4)C(5)C(6)	119·7(1·3)	120·6(1·2)
C(1)C(6)C(5)	121·5(1·1)	119·3(1·1)
C(8)C(7)C(16)	112·8(1·0)	111·4(0·9)
C(8)C(7)C(17)	112·4(1·0)	111·7(0·8)
C(8)C(7)N(1)	98·2(0·9)	100·1(0·7)
C(16)C(7)C(17)	111·1(1·0)	112·2(1·0)
C(16)C(7)N(1)	113·4(0·9)	114·4(0·8)
C(17)C(7)N(1)	108·3(0·9)	106·3(1·0)
C(7)C(8)C(9)	104·0(0·9)	103·9(0·7)
C(8)C(9)C(18)	112·9(0·9)	115·2(0·9)
C(8)C(9)N(2)	108·6(0·9)	108·4(0·7)
C(8)C(9)O(1)	106·5(0·9)	106·2(0·7)
C(18)C(9)N(2)	113·5(0·9)	113·4(0·7)
C(18)C(9)O(1)	107·4(0·9)	106·1(0·7)
N(2)C(9)O(1)	107·6(0·8)	106·9(0·7)
C(11)C(10)C(15)	119·3(1·1)	120·0(0·9)
C(11)C(10)N(2)	117·6(1·0)	116·7(0·7)
C(15)C(10)N(2)	123·0(1·0)	123·3(0·8)
C(10)C(11)C(12)	120·4(1·2)	120·0(0·9)
C(11)C(12)C(13)	121·3(1·3)	120·0(1·0)
C(12)C(13)C(14)	117·6(1·3)	119·2(1·0)
C(13)C(14)C(15)	122·0(1·2)	121·8(1·0)
C(10)C(15)C(14)	119·5(1·1)	119·0(1·0)
C(1)N(1)C(7)	115·9(0·8)	116·6(0·8)
C(1)N(1)O(1)	106·8(0·7)	107·0(0·6)
C(7)N(1)O(1)	100·9(0·7)	101·5(0·6)
C(9)N(2)C(10)	114·9(0·8)	112·7(0·7)
C(9)N(2)O(2)	106·6(0·7)	104·6(0·7)
C(10)N(2)O(2)	108·9(0·7)	109·2(0·6)
C(8)O(1)N(1)	103·2(0·7)	104·2(0·6)

Each of the aromatic rings is planar, but the five-membered ring adopts the half-chair configuration. Table 2 gives the equations of the mean planes of each ring, and the deviations from these planes of relevant atoms.

TABLE 2

Mean planes and deviations (Å) of relevant atoms from them

(*a*) Equations of mean planes through the rings, in the form $lX + mY + nZ + p = 0$,* for (i) type (A) and (ii) type (B)

Plane (1): Ring (A), C(10—(15)

(i) $-0{\cdot}3113X - 0{\cdot}2691Y + 0{\cdot}9114Z - 1{\cdot}650 = 0$

(ii) $-0{\cdot}5724X - 0{\cdot}7851Y + 0{\cdot}2368Z - 4{\cdot}817 = 0$

Plane (2): Ring (B), C(4)—(9), O(1), N(1)

(i) $0{\cdot}1689X + 0{\cdot}4595Y - 0{\cdot}8720Z - 1{\cdot}192 = 0$

(ii) $-0{\cdot}4459X - 0{\cdot}8703Y + 0{\cdot}2095Z - 2{\cdot}270 = 0$

Plane (3): Ring (C), C(1)—(6)

(i) $0{\cdot}3177X + 0{\cdot}9435Y - 0{\cdot}0987Z - 6{\cdot}882 = 0$

(ii) $-0{\cdot}0604X = 0{\cdot}7898Y + 0{\cdot}6104Z - 6{\cdot}670 = 0$

(*b*) Deviations (Å × 10^3) of relevant atoms from the planes, values for type (A) precede those for type (B)

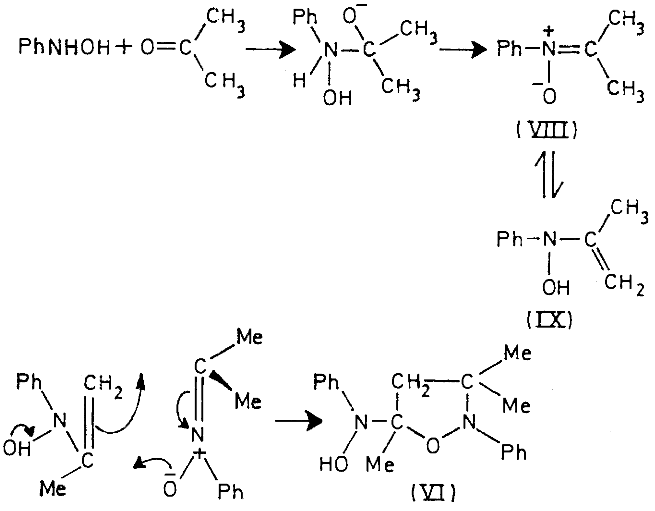
Plane (1): C(10) −4, −2; C(11) 2, 5; C(12) 3, −4; C(13) −6, 1; C(14) 5, 2; C(15) 0, −1; N(2) 62, 14

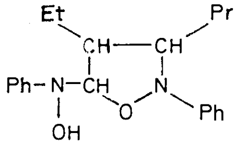
Plane (2): C(7) 260, −226; C(8) −117, 86; C(9) −59, 85; O(1) 236, −242, N(1) −321, 298; C(16) 1754, −1691; C(17) −479, 634; C(18) 1060, −930; N(2) −1426, 1487; C(1) 120, −168

Plane (3): C(1) 15, −2; C(2) 12, 9; C(3) 1, −11; C(4) 7, 6; C(5) −3, 1; C(6) −8, −3; N(1) 34, 23

* *X* is parallel to *a*, *Y* is parallel to *b*, and *Z* is perpendicular to *a* and *b*.

The mechanism of the condensation may be rationalised in terms of the nucleophilic attack by phenylhydroxylamine on acetone to yield (VIII), followed by the 1,3-addition of (VIII) with (IX) to form the product (VI). This mechanism is analogous to that suggested by Kliegel[18] for the condensation of *N*-phenylhydroxylamine with butanal in which compound (X) is formed.





(X)

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[18] W. Kliegel, *Tetrahedron Letters*, 1969, 2627.

The radical formed by ammoniacal silver nitrate oxidation of (VI) is structurally closely related to this precursor, since their i.r. spectra are very similar. The only significant difference is in ν(OH), which is absent in the spectrum of the oxidised product. Furthermore, the mildness of the oxidising agent for the conversion (VI) ⟶ (VII) and of the reducing agent, namely phenylhydrazine, for the reverse process,[3] strongly suggests the structural similarity of these two compounds. Thus, it may be concluded that (VII) represents the structure of the Banfield and Kenyon radical.

The small hyperfine coupling of 0·44 G, assigned by Griffiths and Parlett[15] to a remote methyl group in the incorrect structure (IV), is consistent with coupling to the not-so-remote 5-methyl group in structure (VII).

EXPERIMENTAL

Banfield and Kenyon's method[3] was used for the preparation of (VI) from acetone and *N*-phenylhydroxylamine. The product was washed with acetone and initially recrystallised from ethanol, m.p. 137°. Oxidation with ammoniacal silver nitrate[3] yielded *N*-(3,3,5-trimethyl-2-phenylisoxazolidin-5-yl)phenylaminyl *N*-oxide (VI), m.p. 91 °C, which was recrystallised from light petroleum (b.p. 40—60°). 4-(4-Chloroanilino)-4-methylpentan-2-one (I) was prepared by the action of aqueous hydrochloric acid on (VI), and also by the action of aqueous hydrochloric acid on the product formed between *N*-phenylhydroxylamine and mesityl oxide,[3] b.p. 114° at 0·3 mmHg. The phenylhydrazone was obtained as an oil which was separated, and as an ethereal solution, washed, and dried over sodium sulphate.

TABLE 3

Final fractional co-ordinates (10^4) for both types, with standard deviations in parentheses

	Type (A)			Type (B)		
Atom	*x/a*	*y/b*	*z/c*	*x/a*	*y/b*	*z/c*
C(1)	2934(9)	4879(7)	2521(11)	−1580(7)	1692(16)	5625(6)
C(2)	3851(10)	4674(9)	3448(14)	−923(7)	2609(19)	5151(7)
C(3)	4792(10)	4335(9)	3005(15)	66(8)	2350(21)	5366(8)
C(4)	4853(10)	4173(10)	1708(17)	375(8)	1287(21)	6040(9)
C(5)	3953(11)	4344(10)	788(15)	−277(9)	426(23)	6488(8)
C(6)	3008(9)	4687(9)	1202(12)	−1254(8)	608(19)	6291(7)
C(7)	1915(9)	6380(8)	3141(12)	−3076(7)	3380(16)	5905(6)
C(8)	730(10)	6443(8)	3324(12)	−4039(7)	3486(17)	5378(6)
C(9)	216(9)	5551(8)	2539(11)	−3795(6)	2726(15)	4521(5)
C(10)	−723(8)	3931(8)	2878(11)	−4066(6)	43(15)	3632(5)
C(11)	−39(10)	3117(9)	2858(14)	−3242(7)	−1015(16)	3709(6)
C(12)	−435(12)	2196(10)	2357(15)	−2947(8)	−1971(17)	3006(7)
C(13)	−1512(11)	2070(10)	1868(14)	−3486(9)	−1835(19)	2254(7)
C(14)	−2198(10)	2902(11)	1911(14)	−4300(9)	−776(20)	2190(7)
C(15)	−1822(9)	3819(10)	2397(13)	−4612(7)	172(18)	2876(6)
C(16)	2166(11)	6929(9)	1915(13)	−3214(9)	2717(27)	6798(7)
C(17)	2662(11)	6727(10)	4368(13)	−2529(9)	5144(21)	5901(8)
C(18)	−592(11)	5873(11)	1329(13)	−3956(8)	4021(18)	3805(7)
N(1)	2002(7)	5249(6)	3042(8)	−2597(5)	1843(12)	5477(4)
N(2)	−286(6)	4857(6)	3472(8)	−4325(5)	950(12)	4375(4)
O(1)	1068(5)	5026(5)	2057(7)	−2801(4)	2337(10)	4596(4)
O(2)	−1118(6)	5421(6)	3986(7)	−5338(4)	1433(11)	4277(4)

I.r. spectra were measured as KBr discs by use of a Perkin-Elmer Infracord and a Perkin-Elmer 521 spectrometer. ^{1}H N.m.r. spectra were measured at 90 MHz on a Bruker HX 90 spectrometer.

For the *X*-ray studies two types [(A) and (B)] of crystals of (VI) were obtained in the same batch when crystallised from ethanol. They were both well formed monoclinic prisms, the smaller crystals tending to belong to one form and the larger crystals to the other. In type (A) the prism axis was not the unique axis (*b*) of the unit cell, whereas it was the unique axis in type (B). Unit-cell dimensions for both types were determined from high sin θ reflections on Weissenberg photographs with Cu-K_α radiation. The intensities of the type (A) crystals were collected on a Wooster four-circle diffractometer with Cu-K_α radiation, and those for type (B) crystals on a Hilger linear diffractometer with Mo-K_α radiation. The two sets of data were therefore quite independent, and it is very satisfactory that the structure determinations gave such good agreement.

Crystal Data.—(i) *Type* (A). $C_{18}H_{22}N_2O_2$, $M = 298·4$. Monoclinic, $a = 12·45 \pm 0·01$, $b = 13·25 \pm 0·01$, $c = 10·18 \pm 0·01$ Å, $\beta = 97·7° \pm 0·1$, $U = 1663·6$ Å^3, $D_m = 1·188$, $Z = 4$, $D_c = 1·191$, $F(000) = 640$. Cu-K_α radiation, $\lambda = 1·5418$ Å; μ(Cu-K_α) = 6·31 cm^{-1}. Space group $P2_1/n$. 2317 Independent reflections were measured.

(ii) *Type* (B). $C_{18}H_{22}N_2O_2$, $M = 298·4$. Monoclinic, $a = 13·96 \pm 0·01$, $b = 7·37 \pm 0·01$, $c = 16·14 \pm 0·01$ Å, $\beta = 94·1° \pm 1$, $U = 1656·2$ Å^3, $D_m = 1·193$, $Z = 4$, $D_c = 1·196$, $F(000) = 640$. Mo-K_α radiation, $\lambda = 0·71069$ Å; μ(Mo-K_α) = 0·899 cm^{-1}. Space group $P2_1/n$. 2530 Independent reflections were measured.

Structure Determination.—The structures were determined by the symbolic addition direct method. For type (A), 196 phases were determined and a Fourier synthesis with the corresponding *E* values gave 22 peaks which were consistent with the molecular structure shown in Figure 1. The symbolic addition method produced 200 phases for type (B) and the subsequent *E* Fourier synthesis gave 20 peaks which were accepted as representing C(1)—(15), C(18), N(1), N(2), O(1), and O(2). Another Fourier synthesis using phased *F* values revealed the positions of the missing atoms.

Reflections used to determine the origin in the two structures were: type (A) 961, $\bar{3}94$, 773; and type (B) $\bar{4}31$, $\bar{1}13$, $\bar{1}\bar{1}27$. In each case four reflections were assigned symbols.

Refinement of the atomic co-ordinates and temperature parameters was carried out by the block-diagonal least-squares method and the final values are given in Tables 3 and 4. Calculated hydrogen atom (assuming C–H 1·05 Å) co-ordinates were introduced into the structure-factor calculations in the later stages of the refinement, but were not refined. They were given an isotropic temperature parameter (B) equal to 1·5 times that of the atom to which they were attached. Scattering factors were taken from Berghuis *et al.*[19] The weighting system used was: $w = 1 + \{[(K|F_o| - F^{**})/F^*]^2\}^{-\frac{1}{2}}$ where $F^* = 15$ and $F^{**} = 10$ for type (A) and $F^* = 10$ and $F^{**} = 20$ for type (B).

The final values of R were for (A) 0·093, and for (B) 0·098.

TABLE 4

Final temperature parameters * ($\times 10^4$), with standard deviations in parentheses

(*a*) Type (A)

Atom	b_{11}	b_{12}	b_{13}	b_{22}	b_{23}	b_{33}
C(1)	68(7)	−10(12)	−16(18)	43(6)	22(15)	111(13)
C(2)	90(10)	9(15)	−4(23)	61(7)	29(21)	182(18)
C(3)	72(9)	20(15)	−53(24)	73(8)	−1(24)	221(21)
C(4)	73(9)	−2(15)	44(26)	69(8)	7(26)	285(25)
C(5)	95(10)	−11(16)	62(25)	77(9)	−17(24)	215(21)
C(6)	59(7)	2(13)	15(18)	76(8)	10(19)	135(15)
C(7)	84(9)	−6(13)	14(19)	47(6)	−10(17)	119(14)
C(8)	92(9)	9(14)	29(20)	54(7)	−29(18)	134(15)
C(9)	70(8)	17(12)	2(17)	56(6)	−17(16)	96(12)
C(10)	59(7)	5(12)	42(17)	67(7)	−29(17)	120(13)
C(11)	79(9)	4(14)	51(21)	64(7)	−33(20)	187(18)
C(12)	116(12)	−3(17)	98(26)	72(9)	−60(24)	228(21)
C(13)	108(11)	−39(18)	96(24)	90(9)	−69(24)	196(19)
C(14)	83(10)	−47(17)	22(22)	110(11)	−57(24)	161(17)
C(15)	72(9)	−4(15)	19(21)	82(8)	−32(20)	139(16)
C(16)	102(10)	−18(15)	50(22)	57(7)	21(19)	156(16)
C(17)	123(12)	−49(17)	−22(24)	75(8)	−76(19)	140(16)
C(18)	98(10)	48(18)	17(22)	107(10)	−35(23)	132(16)
N(1)	64(6)	−10(10)	−18(13)	53(5)	−19(13)	88(9)
N(2)	53(5)	22(9)	28(13)	59(5)	−28(13)	106(10)
O(1)	57(5)	−2(8)	−2(11)	63(4)	−33(11)	104(8)
O(2)	80(5)	42(9)	33(12)	77(5)	−37(12)	112(9)

TABLE 4 (*Continued*)

(*b*) Type (B)

Atom	b_{11}	b_{12}	b_{13}	b_{22}	b_{23}	b_{33}
C(1)	42(5)	18(20)	4(7)	204(27)	−24(17)	31(4)
C(2)	44(5)	−2(24)	19(8)	270(31)	−28(22)	47(4)
C(3)	47(6)	−59(27)	24(10)	347(37)	−112(26)	71(6)
C(4)	50(6)	74(28)	−29(11)	397(39)	−123(27)	73(6)
C(5)	70(8)	114(31)	−30(12)	387(41)	−47(28)	59(6)
C(6)	65(7)	74(25)	−3(9)	266(32)	17(21)	42(5)
C(7)	47(5)	33(21)	12(7)	238(28)	−55(17)	33(4)
C(8)	47(5)	45(21)	10(7)	250(29)	−40(17)	31(4)
C(9)	36(4)	20(18)	14(6)	145(23)	−7(15)	31(3)
C(10)	36(4)	−25(18)	18(6)	168(23)	6(15)	29(3)
C(11)	44(5)	−9(20)	19(7)	179(26)	−5(18)	40(4)
C(12)	55(6)	−22(22)	41(8)	203(29)	−37(20)	56(5)
C(13)	71(6)	−67(26)	37(8)	280(33)	−47(21)	40(4)
C(14)	72(7)	−35(29)	7(9)	319(36)	−35(21)	32(4)
C(15)	52(6)	−21(24)	4(8)	275(31)	3(19)	28(4)
C(16)	68(7)	83(36)	17(9)	630(54)	−63(26)	28(4)
C(17)	73(8)	−27(31)	−1(12)	312(37)	−95(25)	60(6)
C(18)	56(6)	47(24)	14(8)	262(29)	79(19)	40(4)
N(1)	38(4)	21(15)	9(5)	185(20)	−7(13)	25(3)
N(2)	29(3)	16(15)	14(5)	177(19)	3(12)	27(3)
O(1)	34(3)	3(13)	12(4)	193(16)	−5(10)	25(2)
O(2)	30(3)	32(13)	8(5)	255(19)	13(12)	35(3)

* In the form $\exp - (h^2b_{11} + hkb_{12} + klb_{13} + k^2b_{22} + klb_{23} + l^2b_{33})$.

Structure factors are listed in Supplementary Publication No. SUP 21036 (12 pp., 1 microfiche).*

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* For details of Supplementary Publications see Notice to Authors No. 7 in *J.C.S. Perkin II*, 1973, Index Issue.

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