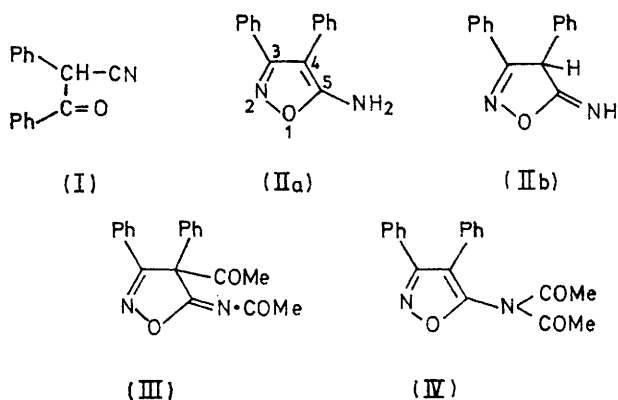


## Crystal and Molecular Structure of 5-Diacetylamino-3,4-diphenylisoxazole

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The structure of (IV) the acetylation product of 5-amino-3,4-diphenylisoxazole has been investigated by spectroscopic methods and determined from three-dimensional X-ray diffraction data. Crystals are orthorhombic, space group  $P2_12_12_1$  with  $Z = 4$  in a unit cell of dimensions:  $a = 9.216(4)$ ,  $b = 9.324(5)$ , and  $c = 19.043(8)$  Å. The crystal structure has been solved by direct methods and refined by block-diagonal least-squares to  $R$  0.063 (1282 independent reflections). The bond lengths of the isoxazole and of the phenyl rings are as expected. The two bonds linking the phenyl rings to the central isoxazole ring, however, are significantly shorter (1.449 and 1.450 Å) than expected for the  $C(sp^2)-C(sp^2)$  bond (1.482 Å). The planes of the phenyl rings and of the two acetyl groups are twisted differently about the bonds linking them to the isoxazole ring.

WALTHER AND SCHICKLER<sup>1</sup> described the compound obtained by the reaction of 2-benzoyl-2-phenylacetone nitrile (I) with hydroxylamine hydrochloride as one of the first 5-aminoisoxazole derivatives (IIa) with the behaviour of the imino-compound (IIb). Compound



(IIa) was also obtained by a new synthetic route, starting from 3-hydroxyamino-2,3-diphenylpropionamide oxime.<sup>2</sup> By acetylation with acetic anhydride from compound (IIa) a diacetyl derivative has been obtained for which structure (III) had been suggested.<sup>1</sup> Under the conditions used, however, C-acetylation is quite unexpected.

**Spectroscopic Investigations.**—The i.r. spectrum of the acetylation product revealed two carbonyl stretching bands at 1746 and 1729  $\text{cm}^{-1}$ . This may be due to the presence of a diacetylamino-group in the molecule, which, however, does not agree with structure (III), the two carbonyl bands being too high for the C-acetyl and acetylimino-group. The n.m.r. spectra recorded in three different solvents ( $\text{CDCl}_3$ ,  $\text{C}_6\text{D}_6$ , and  $\text{CD}_3\text{OD}$ ) showed the two acetyl groups to be equivalent in each case, which supports the existence of the 5-diacetylaminoisoxazole structure (IV). Mass spectrometry revealed that this molecule behaves anomalously. The most intensive fragmentation showed the expulsion of acetic anhydride. This is unexpected in the case of structure (IV), and can be explained only by assuming rearrangement under the fragmentation pathway.<sup>3</sup>

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<sup>1</sup> R. Walther and P. G. Schickler, *J. prakt. Chem.*, 1897, **55**, (2), 305.

<sup>2</sup> K. Harsányi, K. Takács, and K. Horváth, *Conf. Hungarian Chem. Soc.*, Pécs, 1973.

As the spectral results were not free from contradictions it seemed reasonable to investigate the structure of the diacetyl molecule by X-ray diffraction.

### EXPERIMENTAL

**Crystal Data.**— $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_3$ ,  $M = 320.354$ . Orthorhombic,  $a = 9.216(4)$ ,  $b = 9.324(5)$ ,  $c = 19.043(8)$  Å,  $U = 1636.37$  Å<sup>3</sup>,  $D_m = 1.297$  (by flotation),  $Z = 4$ ,  $D_c = 1.301$ ,  $F(000) = 672$ ,  $\text{Cu-K}\alpha$  radiation,  $\lambda = 1.5418$  Å,  $\mu(\text{Cu-K}\alpha) = 5.69$   $\text{cm}^{-1}$ . Space group  $P2_12_12_1$  from systematic absences.

**Crystallographic Measurements.**—Cell dimensions were determined from precession photographs. Reflections for the reciprocal layers  $h = 0-9$  were collected on a semi-automatic two-circle Stoe-Güttinger single-crystal diffractometer operating with the equi-inclination Weissenberg procedure. Intensities were measured by rotation about the  $b$  axis for  $2\theta$  0–116° and evaluated according to a procedure reported elsewhere.<sup>4</sup> The size of the crystal (0.3 mm) satisfied the condition  $\mu R < 0.3$  and no absorption correction was made. After Lorentz-polarization correction the absolute scale and temperature factor ( $B$  4.640 Å<sup>2</sup>) were determined by a Wilson plot.

**Determination of the Structure.**—The phase problem was solved by direct methods by use of the MULTAN program.<sup>5</sup> Starting with the  $E$  values of 171 reflections ( $E \geq 1.45$ ) taking 1200 phase relationships, sign-accepted with probability  $> 0.80$ , and also using three origin-fixing reflections, two starting reflections, of which one was used for the enantiomorph, eight sets of phases were obtained. Out of these eight, two were found with the greatest but not significantly different ABSFOM values. In order to obtain only one, the most probable solution, a third starting reflection was collected. For this purpose, the last reflection in the convergence map with no contributors was chosen. This reflection with  $\pm \frac{\pi}{4}$  and  $\pm \frac{3\pi}{4}$  phases was attached to the former starting reflections with their phases in the previous two most probable phase sets. The repetition of the tangent formula with eight possible phase combinations resulted in only one set of phases with greatest ABSFOM. The  $E$  map, calculated with this phase set, at once revealed the peaks of all non-hydrogen atoms.

The initial structure-factor agreement, based on the  $E$  map co-ordinates and overall temperature factor (from

<sup>3</sup> J. Tamás, personal communication.

<sup>4</sup> K. Sasvári, K. Simon, R. Bognár, and S. Makleit, *Acta Cryst.*, 1974, **B30**, 634.

<sup>5</sup> P. Main, M. M. Woolfson, and G. Germain, MULTAN, Program for Automatic Solution of Crystal Structures, 1971, University of York, York, England.

TABLE 1

Final fractional co-ordinates ( $\times 10^4$ ) and anisotropic thermal parameters\* ( $\times 10^4$ ) for non-hydrogen atoms, with estimated standard deviations in parentheses

Atom	$x$	$y$	$z$	$b_{11}$	$b_{22}$	$b_{33}$	$b_{12}$	$b_{13}$	$b_{23}$
O(1)	-902(6)	2622(5)	173(2)	204(7)	160(6)	33(1)	85(12)	-7(5)	-26(5)
N(2)	-431(7)	2093(6)	826(3)	174(8)	175(7)	30(1)	42(14)	-13(6)	-27(6)
C(3)	280(7)	877(7)	693(3)	122(7)	154(8)	28(2)	25(15)	-9(7)	-24(6)
C(4)	312(7)	570(7)	-42(3)	149(8)	134(7)	25(2)	7(14)	-11(7)	-20(6)
C(5)	-451(8)	1682(7)	-321(3)	156(8)	145(8)	28(2)	18(15)	13(7)	-10(6)
C(6)	857(7)	20(7)	1264(3)	157(8)	159(8)	23(1)	-17(17)	-13(7)	-23(6)
C(7)	142(8)	-71(9)	1909(3)	164(9)	200(10)	27(2)	-36(19)	4(7)	-13(8)
C(8)	688(9)	-932(10)	2435(3)	203(11)	231(12)	26(2)	-25(22)	21(8)	4(8)
C(9)	1934(9)	-1729(8)	2337(3)	190(11)	187(10)	34(2)	-65(19)	-20(8)	18(8)
C(10)	2655(8)	-1638(9)	1702(3)	175(10)	225(11)	30(2)	58(20)	-35(8)	19(8)
C(11)	2126(7)	-759(8)	1175(3)	154(9)	202(10)	28(2)	-3(18)	1(7)	-21(8)
C(12)	881(7)	-661(7)	-416(3)	152(8)	144(7)	22(1)	34(16)	-5(6)	0(6)
C(13)	382(8)	-2044(7)	-266(3)	154(8)	153(8)	29(2)	26(16)	-16(7)	-11(6)
C(14)	913(9)	-3220(8)	-619(4)	190(10)	165(9)	38(2)	59(18)	-44(8)	-29(8)
C(15)	1967(9)	-3048(8)	-1127(4)	189(10)	182(9)	39(2)	72(19)	-17(9)	-47(8)
C(16)	2504(8)	-1685(9)	-1276(4)	174(10)	214(11)	34(2)	28(20)	18(8)	-21(8)
C(17)	1973(8)	-497(8)	-924(3)	179(9)	159(8)	28(2)	52(17)	6(7)	-16(7)
N(18)	-898(6)	1949(6)	-1020(3)	164(7)	144(6)	27(1)	36(13)	-8(6)	2(6)
C(19)	-2042(8)	1095(9)	-1304(4)	173(9)	216(11)	34(2)	54(21)	-32(8)	-9(8)
C(20)	-9(8)	2951(7)	-1395(4)	184(10)	148(8)	36(2)	83(17)	22(8)	-5(7)
O(21)	-2231(7)	1051(8)	-1927(3)	268(9)	337(12)	37(2)	-147(20)	-52(7)	0(8)
C(22)	-2888(9)	279(9)	-795(4)	187(11)	211(11)	49(2)	-18(21)	-13(10)	24(10)
O(23)	1033(6)	3425(6)	-1102(3)	222(8)	162(6)	50(2)	-49(14)	-1(7)	11(6)
C(24)	-448(11)	3365(10)	-2134(4)	271(14)	224(12)	37(2)	88(25)	20(10)	51(9)

\* Defined by:  $T = \exp -(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{13}hl + b_{23}kl)$ .

the Wilson plot), was 27.4%. Refinement proceeded to  $R$  13.3% after 3 cycles of block-diagonal least-squares with isotropic, and to  $R$  8.3% after three subsequent cycles of least-squares with anisotropic temperature factors. The function minimized was  $\Phi = \sum_h w_h(F_o -$

$\frac{1}{G} |F_c|)^2$ , using the weighting factor of Cruickshank<sup>6</sup> where  $w_h = 1/(4.0 + F + 0.04F^2)$ . After refinement as before the positions of all 16 hydrogen atoms were obtained from the difference Fourier map.

TABLE 2

Final fractional co-ordinates ( $\times 10^3$ ) and isotropic thermal parameters ( $\text{\AA}^2$ ) of the hydrogen atoms, with estimated standard deviations in parentheses

Atom	$x$	$y$	$z$	$B$
H(25)	-79(11)	52(11)	192(4)	5.8
H(26)	24(12)	-96(13)	288(5)	8.6
H(27)	245(10)	-233(10)	275(4)	4.4
H(28)	348(11)	-233(12)	168(5)	7.0
H(29)	267(10)	-73(10)	70(4)	4.2
H(30)	-28(9)	-222(8)	5(4)	4.2
H(31)	51(10)	-423(10)	-53(4)	4.7
H(32)	251(10)	-385(11)	-137(5)	8.7
H(33)	335(12)	-148(12)	-165(5)	5.8
H(34)	237(11)	40(11)	-103(5)	7.6
H(35)	-300(11)	98(11)	-38(5)	8.8
H(36)	-248(13)	-70(13)	-59(6)	10.2
H(37)	-382(15)	3(14)	-109(5)	11.1
H(38)	-2(17)	402(17)	-233(6)	13.9
H(39)	-19(16)	251(15)	-243(6)	8.8
H(40)	-148(13)	365(13)	-219(5)	8.9

When all the hydrogen atoms were introduced into the structure-factor calculation, two cycles of refinement of all atoms, hydrogen atoms with isotropic temperature factors, gave  $R$  0.057 for the observed (1160) and  $R$  0.063 for all (1282) reflections. Final atomic para-

\* See Notice to Authors No. 7 in *J.C.S. Perkin II*, 1973, Index issue.

eters are given in Tables 1 and 2. Structure amplitudes calculated from the final atomic parameters are listed in Supplementary Publication No. SUP 21053 (2 pp.).\* Atomic scattering factors were taken from ref. 7.

## DISCUSSION

*Description of the Molecule.*—The atomic numbering system used is shown in Figure 1. Thus the crystal

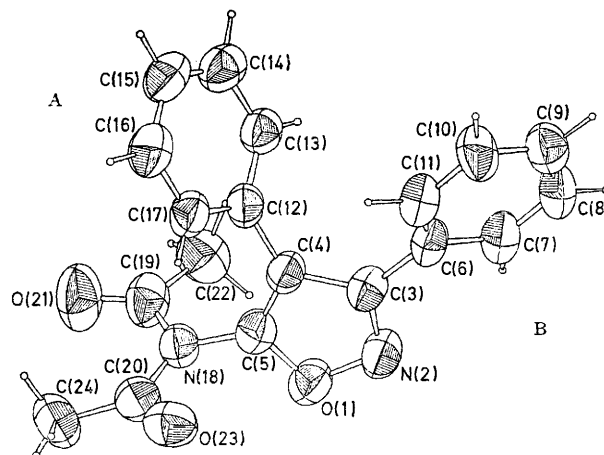


FIGURE 1 Perspective view of the molecule with non-hydrogen atoms represented by their thermal vibration ellipsoids

structure analysis justified the correctness of structure (IV). The conformation of the molecule can be characterized by six planes (Table 3). Plane (1), formed by the isoxazole ring and the three nearest-neighbouring

<sup>6</sup> D. W. J. Cruickshank 'Computing Methods and the Phase Problem in X-Ray Crystal Analysis,' Pergamon Press, Oxford, 1961.

<sup>7</sup> 'International Tables for X-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1962.

atoms, intersects the planes of the two phenyl rings [(2) and (3)] along bonds C(3)-C(6) and C(4)-C(12), respectively. About these intersections the planes (2) and (3) are twisted with torsion angles 35.7 and/or 61.7°, relative to plane (1), resulting in an inclination angle of 122.2° for the phenyl planes (2) and (3). The atoms of the diacetyl-amino-group form a rather deformed plane (4) with maximum deviation 0.25 Å; it is therefore reasonable to regard separately the two *N*-acetyl groups forming planes (5) and (6). These planes have a common intersection with plane (1) along the C(5)-N(18) bond, and the (5) and (6) planes are

TABLE 3

(a) Equations of best least-squares planes in the molecule in the form  $AX + BY + CZ = D$ , where  $X$ ,  $Y$ , and  $Z$  are co-ordinates (Å) referred to crystal axes. Distances (Å) of relevant atoms from the planes are given in square brackets

	<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>
Plane (1): O(1), N(2), C(3)—(6), C(12), N(18)	0.8572	0.5035	-0.1086	0.4724
[O(1) 0.01, N(2) 0, C(3) 0.02, C(4) 0.05, C(5) 0.03, C(6) -0.05, C(12) 0, N(18) -0.06, C(7) -0.79, C(11) 0.61, C(13) -1.08, C(17) 1.04, C(19) -0.13, C(20) 1.19, C(22) -2.46, C(24) 1.19]				
Plane (2): C(3), C(6)—(11)	0.5367	0.7722	0.3401	1.2437
[C(3) -0.02, C(6) 0.01, C(7) 0.01, C(8) 0, C(9) -0.02, C(10) -0.01, C(11) 0.02, N(2) 0.59, C(4) -0.71, H(25) -0.01, H(26) 0.04, H(27) 0.07, H(28), -0.11 H(29) 0]				
Plane (3): C(4), C(12)—(17)	0.7050	-0.1088	0.7008	0.0921
[C(4) 0, C(12) -0.01, C(13) 0.01, C(14) 0, C(15) -0.01, C(16) 0, C(17) 0.01, C(3) 0.92, C(5) -0.98, C(6) 2.15, C(7) 2.56, C(11) 2.93, N(18) -2.23, C(19) 3.27, C(20) 2.26, H(22) 3.06, H(30) 0.02, H(31) -0.04, H(32) 0.10, H(33) 0.03, H(34) 0.03]				
Plane (4): C(5), N(18), C(19), C(20), O(21), C(22), O(23), C(24)	-0.6285	0.7324	0.2620	1.2994
[C(5) -0.05, N(18) 0.04, C(19) -0.02, C(20) 0.03, O(21) -0.25, C(22) 0.17, O(23) -0.11, C(24) 0.19, O(1) 1.10, C(4) -1.11, C(6) -1.15, C(7) -0.48, C(11) -2.46, C(12) -2.47, C(13) -3.05, C(17) -3.24]				
Plane (5): C(5), N(18), C(19), O(21), C(22)	-0.6344	0.7690	0.0790	1.7722
[C(5) -0.10, N(18) 0.13, C(19) 0.03, O(21) -0.08, C(22) 0.03, C(20) 0.29, O(23) 0.16, C(24) 0.61]				
Plane (6): C(5), N(18), C(20), O(23), C(24)	-0.5504	0.7529	0.3610	1.1213
[C(5) 0.02, N(18) -0.02, C(20) -0.01, O(23) -0.01, C(24) 0.02, C(19) -0.24, O(21) -0.59, C(22) -0.06]				
(b) Angles (deg.) between planes				
(1)-(2)	144.3	(1)-(5)	83.3	
(1)-(3)	118.3	(1)-(6)	82.9	
(1)-(4)	78.6	(5)-(6)	168.6	
(2)-(3)	122.2			

twisted about this intersection in opposite directions with angles of 83.3 and 82.9°, respectively, relative to plane (1), resulting in an inclination angle of 168.6° for planes (5) and (6). Besides this inclination, the

<sup>8</sup> S. Biagini, M. Cannas, and G. Marongiu, *Acta Cryst.*, 1969, **B25**, 2108.

<sup>9</sup> M. Sundaralingam and G. A. Jeffrey, *Acta Cryst.*, 1962, **15**, 1035.

acetyl groups are also in asymmetric positions relative to plane (1) (Figure 1). This might be the reason for the two different carboxy bands found in the i.r. spectrum.

Atomic bond lengths and bond angles in the molecule are given in Tables 4 and 5. The mean of C-H bond

TABLE 4

Bond lengths (Å) with estimated standard deviations in parentheses

O(1)-N(2)	1.406(8)	C(10)-C(11)	1.384(10)
O(1)-C(5)	1.351(8)	C(12)-C(13)	1.399(9)
N(2)-C(3)	1.334(9)	C(12)-C(17)	1.404(9)
C(3)-C(4)	1.429(9)	C(13)-C(14)	1.376(10)
C(3)-C(6)	1.450(9)	C(14)-C(15)	1.380(11)
C(4)-C(5)	1.361(9)	C(15)-C(16)	1.393(11)
C(4)-C(12)	1.449(9)	C(16)-C(17)	1.384(11)
C(5)-N(18)	1.415(9)	N(18)-C(19)	1.428(10)
C(6)-C(7)	1.396(9)	N(18)-C(20)	1.433(9)
C(6)-C(11)	1.387(9)	C(19)-O(21)	1.200(10)
C(7)-C(8)	1.379(11)	C(19)-C(22)	1.458(11)
C(8)-C(9)	1.380(11)	C(20)-O(23)	1.195(9)
C(9)-C(10)	1.382(10)	C(20)-C(24)	1.514(12)

TABLE 5

Bond angles (°), with estimated standard deviations in parentheses

N(2)-O(1)-C(5)	107.1(5)	C(6)-C(11)-C(10)	121.2(6)
O(1)-N(2)-C(3)	106.4(5)	C(4)-C(12)-C(13)	120.7(6)
N(2)-C(3)-C(4)	111.5(6)	C(4)-C(12)-C(17)	120.8(6)
N(2)-C(3)-C(6)	120.4(6)	C(13)-C(12)-C(17)	118.5(6)
C(4)-C(3)-C(6)	128.1(6)	C(12)-C(13)-C(14)	121.2(6)
C(3)-C(4)-C(5)	102.7(5)	C(13)-C(14)-C(15)	120.0(7)
C(3)-C(4)-C(12)	130.4(6)	C(14)-C(15)-C(16)	119.9(7)
C(5)-C(4)-C(12)	126.8(6)	C(15)-C(16)-C(17)	120.4(7)
O(1)-C(5)-C(4)	112.4(6)	C(12)-C(17)-C(16)	120.0(6)
O(1)-C(5)-N(18)	116.8(6)	C(5)-N(18)-C(19)	118.2(6)
C(4)-C(5)-N(18)	130.7(6)	C(5)-N(18)-C(20)	114.6(5)
C(3)-C(6)-C(7)	121.3(6)	C(19)-N(18)-C(20)	126.7(6)
C(3)-C(6)-C(11)	120.4(6)	N(18)-C(19)-O(21)	120.1(7)
C(7)-C(6)-C(11)	118.3(6)	N(18)-C(19)-C(22)	115.7(7)
C(6)-C(7)-C(8)	120.1(7)	O(21)-C(19)-C(22)	124.2(7)
C(7)-C(8)-C(9)	121.3(7)	N(18)-C(20)-O(23)	117.9(6)
C(8)-C(9)-C(10)	119.0(7)	N(18)-C(20)-C(24)	118.5(6)
C(9)-C(10)-C(11)	120.1(7)	O(23)-C(20)-C(24)	123.7(7)

lengths is 1.00 Å, maximum deviation 0.12 Å. The phenyl rings are therefore regular and congruent with each other. The mean C-C bond lengths of the two phenyl rings (A and B) are 1.385 and 1.389 Å, with maximum deviations of 0.011 and 0.015 Å, respectively, from individual bond lengths in the two rings. These deviations are <1.5σ. The angles of the phenyl rings are close to 120° with maximum deviations of 1.5 and 1.7° for the two rings.

The conformation of the isoxazole ring, considering bond lengths and bond angles, is comparable with those reported previously<sup>8-11</sup> (Table 6). Bond lengths and also angles differ according to the structure, but alterations for bond lengths are within 3σ and only a little greater for angles. The same is true if we compare the isoxazole rings of the two independent molecules in the same structure [*e.g.* in columns (6a) and (6b), and (7a) and (7b) in Table 6].

The C-C single bonds linking the phenyl rings to the

<sup>10</sup> L. Brehm, personal communication.

<sup>11</sup> S. Biagini, M. Cannas, and G. Marongiu, *Acta Cryst.*, 1969, **B25**, 730.

isoxazole ring (1.450 and 1.449 Å) are significantly shorter than the mean value (1.482 Å) reported<sup>12</sup> for the C(sp)<sup>2</sup>-C(sp)<sup>2</sup> bond lengths, or the bonds (1.470 and 1.485 Å) linking phenyl rings to the five-membered ring of the indole system in 2,3-diphenylindole.<sup>13</sup>

In (IV), at the sites of linkage of substituents to the isoxazole ring, the enlarged bond angles C(4)-C(3)-C(6)

(from rigid-body analysis) with those observed (from thermal parameters), there was satisfactory agreement for the rigid-body assumption for only one fraction of the molecule, namely the five-membered isoxazole ring.

The spatial packing of the molecules in the crystal structure corresponds to the molecular close-packing

TABLE 6

A comparison of bond lengths (Å) and angles (°) for the isoxazole ring in different crystal structures. Column headings are defined in footnote; (a) and (b) distinguish two independent molecules in the unit cell

	(1)	(2)	(3)	(4)	(5)	(6a)	(6b)	(7a)	(7b)
O(1)-N(2)	1.406(8)	1.417(5)	1.412(7)	1.427(5)	1.425(4)	1.440(4)	1.427(4)	1.422(6)	1.421(6)
N(2)-C(3)	1.334(9)	1.313(7)	1.344(9)	1.331(5)	1.316(4)	1.323(4)	1.311(4)	1.322(7)	1.321(7)
C(3)-C(4)	1.429(9)	1.408(6)	1.425(8)	1.440(5)	1.435(4)	1.427(4)	1.437(4)	1.428(7)	1.394(6)
C(4)-C(5)	1.361(9)	1.372(6)	1.394(8)	1.338(6)	1.322(4)	1.340(5)	1.329(4)	1.341(7)	1.372(5)
C(5)-O(1)	1.351(9)	1.372(6)	1.357(9)	1.358(5)	1.349(4)	1.343(4)	1.347(4)	1.346(6)	1.342(6)
C(5)-O(1)-N(2)	107.1(5)	109.0(4)	110.5	108.3(3)	107.6(3)	108.1(2)	107.0(2)	108.4(3)	106.9(4)
O(1)-N(2)-C(3)	106.4(5)	105.2(4)	104.6	106.1(3)	106.1(2)	105.1(2)	107.2(3)	105.1(4)	105.6(4)
N(2)-C(3)-C(4)	111.5(6)	112.4(4)	111.9	109.5(4)	109.8(2)	110.9(3)	109.1(3)	111.2(4)	112.9(4)
C(3)-C(4)-C(5)	102.7(5)	105.2(4)	104.4	106.2(4)	105.7(2)	105.3(2)	106.0(3)	104.6(4)	103.0(4)
C(4)-C(5)-O(1)	112.4(6)	108.1(4)	108.5	109.9(4)	110.8(2)	110.7(3)	110.8(3)	110.7(4)	111.7(4)

(1) Present work; (2) 3-hydroxy-5-phenylisoxazole ( $\beta$ -form), C<sub>9</sub>H<sub>7</sub>NO<sub>2</sub>, ref. 8; (3) 3-benzoyl-2,1-benzisoxazole (2-phenylisoxatogen), C<sub>14</sub>H<sub>9</sub>NO<sub>2</sub>, ref. 9; (4) Muscimol, 5-aminomethyl-3-hydroxyisoxazole, C<sub>4</sub>H<sub>6</sub>N<sub>2</sub>O<sub>2</sub>, ref. 10; (5) Homomuscimol, 5-(2-aminoethyl)-3-hydroxyisoxazole, C<sub>5</sub>H<sub>8</sub>N<sub>2</sub>O<sub>2</sub>, ref. 10; (6) Muscimol, 5-aminomethyl-3-hydroxyisoxazole, C<sub>4</sub>H<sub>6</sub>N<sub>2</sub>O<sub>2</sub>·0.5H<sub>2</sub>O, ref. 10; (7) 3,4'-bi-isoxazole, C<sub>6</sub>H<sub>4</sub>N<sub>2</sub>O<sub>2</sub>, ref. 11.

and C(4)-C(5)-N(18) (128.1 and/or 130.7°), as well as the torsion of the phenyl rings and diacetyl-amino-group relative to the isoxazole ring (Table 3) may be due to a strained condition. Similar strain and torsion can also be found in 2,3-diphenylindole,<sup>13</sup> as well.

The thermal vibration ellipsoids of the non-hydrogen atoms with their root-mean-square amplitudes have been determined and represent the corresponding atoms in Figure 1. The thermal motion of the molecule was also analysed by rigid-body assumption for the

where the molecules are held together by van der Waals forces. Shortest intermolecular H...H and C...H

TABLE 7  
Shortest intermolecular distances (Å) in the spatial packing of the molecules in crystal lattice

H(25) ... H(27 <sup>I</sup> )	2.595 *	C(7) ... H(32 <sup>II</sup> )	2.821 *
H(25) ... H(32 <sup>II</sup> )	2.452	C(8) ... H(33 <sup>IV</sup> )	2.982
H(26) ... H(37 <sup>III</sup> )	2.528	C(10) ... H(39 <sup>IV</sup> )	2.974
H(27) ... H(39 <sup>IV</sup> )	2.562	C(14) ... H(36 <sup>V</sup> )	2.917
H(28) ... H(39 <sup>IV</sup> )	2.321	C(20) ... H(27 <sup>V</sup> )	2.923
H(29) ... H(31 <sup>V</sup> )	2.644	C(24) ... H(27 <sup>VI</sup> )	2.937
Mean	2.517	Mean	2.926

\* Sum of van der Waals radii give H...H 2.5, C...H 2.9. Roman numeral superscripts denote the following translation of the atom, relative to the reference molecule at  $x, y, z$ :

$$\begin{array}{ll} \text{I} & -x, \frac{1}{2} + y, \frac{1}{2} - z \\ \text{II} & -\frac{1}{2} + x, -\frac{1}{2} - y, -z \\ \text{III} & -\frac{1}{2} - x, -y, \frac{1}{2} + z \\ \text{IV} & \frac{1}{2} - x, -y, \frac{1}{2} + z \\ \text{V} & \frac{1}{2} + x, -\frac{1}{2} - y, -z \\ \text{VI} & \frac{1}{2} - x, -y, -\frac{1}{2} + z \end{array}$$

whole molecule as well as of its different fractions. Comparing  $U$  tensors of the individual atoms calculated

<sup>12</sup> M. J. S. Dewar and H. N. Schmeising, *Tetrahedron*, 1960, **11**, 96.

<sup>13</sup> B. Schmelter, H. Bradaczek, and P. Luger, *Acta Cryst.*, 1973, **B29**, 971.

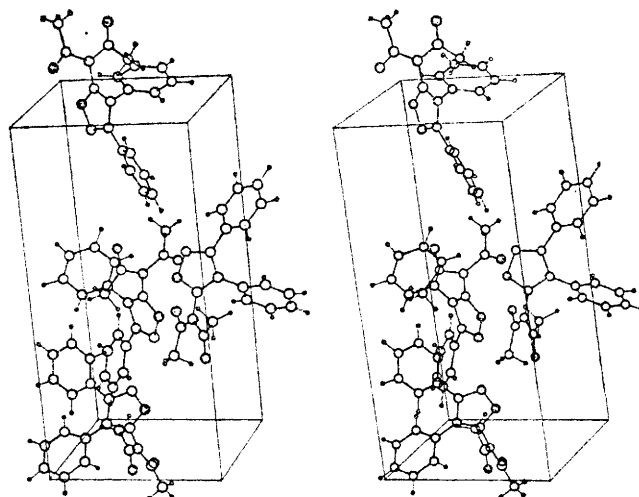


FIGURE 2 Stereoscopic projection of the unit cell as viewed down a direction differing from the  $a$  crystal axis by twists of 20 and  $-20^\circ$  around  $b$  and  $c$  crystal axis, respectively

distances are given in Table 7. The molecular packing is illustrated by a stereoscopic projection of the unit cell (Figure 2).<sup>14</sup>

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<sup>14</sup> C. K. Johnson, program ORTEP, Oak Ridge National Laboratory Report ORNL 3794, Oak Ridge, Tennessee, U.S.A.