

température forts dans le composé sélénié [B isotopes équivalents: $O(2) = 9,1$; $O(5) = 8,0 \text{ \AA}^2$] et d'autre part une variation décroissante des températures de fusion lorsque X passe de S à O (respectivement 98 et 79°). Le point de fusion du composé sélénié n'est pas mesurable du fait de sa décomposition et la valeur indiquée dans la littérature (458 K décomposition) (Butler *et al.*, 1975) ne sous semble pas correcte.

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Structure of a 3-Acetylindeneindolin-2-one Dimer

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Abstract. $C_{22}H_{18}N_2O_4$, triclinic, $P\bar{1}$, $a = 12.561$ (6), $b = 8.857$ (4), $c = 8.644$ (4) \AA , $\alpha = 80.39$ (5), $\beta = 103.72$ (7), $\gamma = 107.28$ (7)°, $Z = 2$. The final R value is 0.05 for 1701 observed reflexions. The molecule adopts a folded conformation.

Introduction. On heating (*E*)-3-acetylindeneindolin-2-one (I) at 100°C in toluene solutions for 24 hours, a dimeric product (II) and a novel regioisomeric dimer (III) are produced. The chemical structure of compound (II) has been determined by Banfield, Johnson & Katner (1966), while that of the dimer (III) has only been proposed, on the basis of proton and ^{13}C magnetic resonance spectroscopic analyses, as shown in Fig. 1 (Kubo, Nakai, Nozoye, Itai & Iitaka, 1978) and the stereochemistry of the compound remained undetermined.

The present X-ray study has been undertaken in order to elucidate the configuration and conformation of (III).

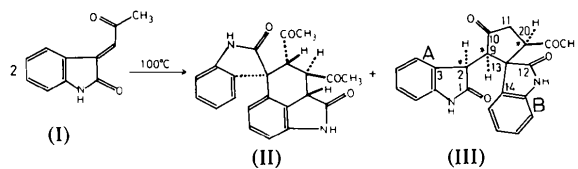


Fig. 1. Reaction scheme.

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The compound (III) was recrystallized from methanol solution as colorless prisms, m.p. $267\text{--}270^\circ\text{C}$. Intensity data were measured on a Philips PW 1100 automatic diffractometer using $\text{Cu } K\alpha$ radiation monochromated by a graphite plate. The $2\theta\text{--}\theta$ scanning technique was adopted with a scan speed of 0.1° s^{-1} in θ . Three standard reflexions were measured every two hours, but no significant change was observed. Background was measured at each end of the scan range for half the total scan time. A total of 1707 non-zero independent reflexions were collected out of 2600 possible reflexions within a 2θ angle of 120° . Intensities were then corrected for Lorentz and polarization factors, but no correction was applied for either absorption or extinction. The structure was solved by the direct method using *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974).

All the 28 non-hydrogen atoms were located on an E map and their parameters were refined by several cycles of block-diagonal least-squares calculations. H atoms were obtained from a difference electron density map. Subsequent refinements assuming anisotropic thermal parameters for non-hydrogen atoms and isotropic ones for H atoms gave the final R factor of 0.05. The weighting system was: $\sqrt{w} = 1.0$ when $F_o < 15.0$; $\sqrt{w} = 15.0/F_o$ when $F_o \geq 15.0$. Atomic scattering factors for C, N and O atoms were those given in *International Tables for X-ray Crystallography* (1962) as SX-6, 7 and 8 and for H atoms those

given by Stewart, Davidson & Simpson (1965). The final atomic coordinates are listed in Table 1.*

Discussion. The molecular structure is shown in Fig. 2 by an *ORTEP* drawing (Johnson, 1965), along with atomic numbering. The bond lengths and valency angles are shown in Fig. 3. The standard deviations are

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33746 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates ($\times 10^4$ for non-hydrogen atoms and $\times 10^3$ for hydrogen atoms)

Estimated standard deviations are given in parentheses.

	<i>x</i>	<i>y</i>	<i>z</i>
O(1)	5725 (2)	8613 (3)	4016 (3)
O(2)	3667 (3)	5602 (4)	-677 (4)
O(3)	4541 (2)	11104 (3)	1786 (3)
O(4)	943 (3)	9016 (4)	-2310 (4)
N(1)	4383 (3)	6727 (4)	5226 (3)
N(2)	3152 (3)	9857 (4)	3242 (4)
C(1)	4907 (3)	7443 (5)	3996 (5)
C(2)	4305 (3)	6513 (5)	2539 (4)
C(3)	3417 (3)	5176 (4)	3199 (4)
C(4)	2652 (4)	3870 (5)	2536 (5)
C(5)	1937 (4)	2784 (5)	3472 (6)
C(6)	2029 (4)	3003 (5)	5045 (6)
C(7)	2812 (4)	4277 (5)	5731 (5)
C(8)	3493 (3)	5359 (5)	4800 (5)
C(9)	3951 (3)	7620 (4)	1067 (4)
C(10)	3509 (3)	6822 (5)	-465 (5)
C(11)	2825 (3)	7783 (5)	-1663 (5)
C(12)	3703 (3)	10022 (4)	2028 (4)
C(13)	3108 (3)	8613 (4)	1027 (4)
C(14)	2098 (3)	7774 (4)	1813 (4)
C(15)	1185 (3)	6472 (5)	1440 (5)
C(16)	381 (3)	5972 (5)	2417 (6)
C(17)	477 (3)	6765 (5)	3703 (5)
C(18)	1378 (3)	8092 (5)	4070 (5)
C(19)	2179 (3)	8559 (4)	3116 (4)
C(20)	2808 (3)	9160 (5)	-817 (4)
C(21)	1723 (4)	9676 (5)	-1332 (5)
C(22)	1674 (5)	11086 (6)	-625 (6)
H(N1)	462 (5)	708 (6)	622 (7)
H(N2)	341 (6)	1062 (8)	411 (8)
H(C2)	488 (4)	611 (6)	217 (6)
H(C4)	254 (4)	375 (6)	138 (6)
H(C5)	139 (3)	184 (4)	298 (5)
H(C6)	155 (3)	222 (5)	576 (5)
H(C7)	294 (3)	448 (4)	682 (5)
H(C9)	464 (3)	838 (4)	88 (4)
H(C11)	209 (3)	717 (4)	-188 (5)
H'(C11)	317 (4)	823 (5)	-263 (5)
H(C15)	111 (4)	590 (5)	55 (5)
H(C16)	-25 (5)	507 (7)	215 (7)
H(C17)	-10 (3)	640 (5)	436 (5)
H(C18)	145 (3)	871 (4)	495 (4)
H(C20)	345 (3)	1009 (4)	-102 (5)
H(C22)	161 (4)	1079 (6)	53 (6)
H'(C22)	107 (4)	1146 (6)	-111 (6)
H''(C22)	250 (6)	1191 (8)	-53 (8)

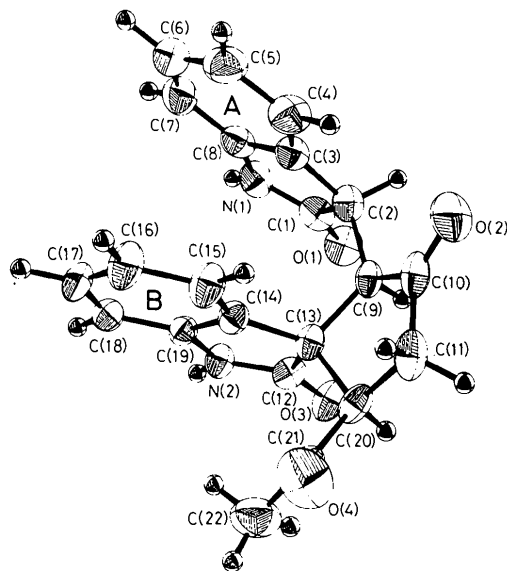


Fig. 2. An *ORTEP* drawing (Johnson, 1965) along with the atomic numbering.

estimated as $\sigma(\text{C}-\text{C}) = 0.005$, $\sigma(\text{C}-\text{H}) = 0.04$ Å and $\sigma(\text{C}-\text{C}-\text{C}) = 0.4^\circ$, $\sigma(\text{C}-\text{C}-\text{H}) = 3^\circ$ and $\sigma(\text{H}-\text{C}-\text{H}) = 5^\circ$. As expected, a cyclopentanone ring is formed by dimerization, to which are attached the oxindole ring *B* at the spiranic C atom C(13) and the oxindole ring *A* and an acetyl group. The latter two substituents are linked to the cyclopentanone ring on the same side of

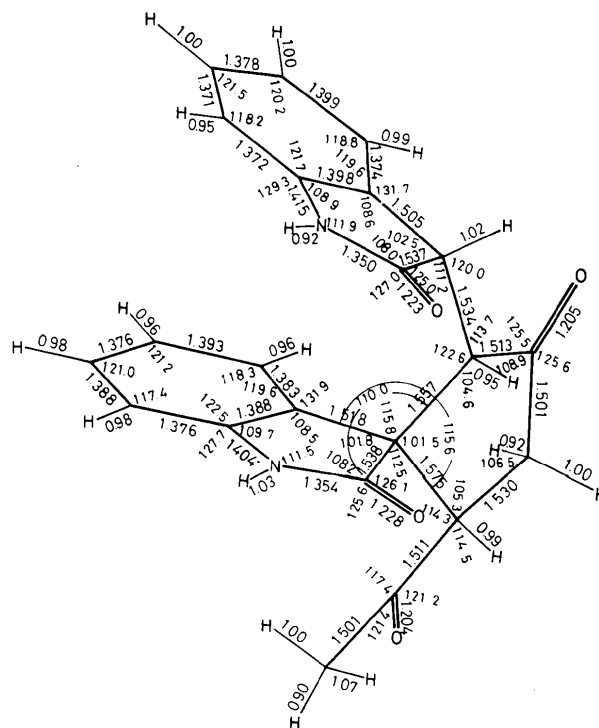


Fig. 3. Bond lengths (Å) and valency angles ($^\circ$).

the ring. The stereochemistry about the C(2) and C(9) atoms conforms to the *threo* configuration. As a result of the *gauche-gauche* conformation of the C(2)–C(3) bond with respect to the C(9)–C(13) and C(9)–C(10) bonds, the oxindole ring *A* comes close to *B*, their planes facing each other. The dihedral angle between these two planes is 21.2° . Thus the molecule adopts a folded conformation. The Newman projection along the C(2)–C(9) bond is illustrated in Fig. 4.

This conformation, although it seems to be less stable, may be stabilized by the intramolecular as well as intermolecular interactions between the two oxindole groups. Fig. 5 shows the stacking and interactions of the oxindole groups viewed perpendicular to the plane of oxindole *A*. The closest intramolecular interatomic distances between *A* and *B* are 3.156, 3.151, 3.143 and 3.252 Å of O(1)–O(3), C(1)–C(12), C(3)–C(14) and C(4)–C(15), respectively. The mean separation of the

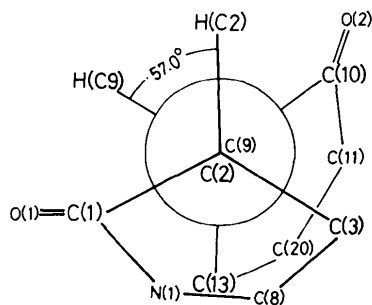


Fig. 4. Newman projection down the C(2)–C(9) bonds, along with the partial structure of the molecule.

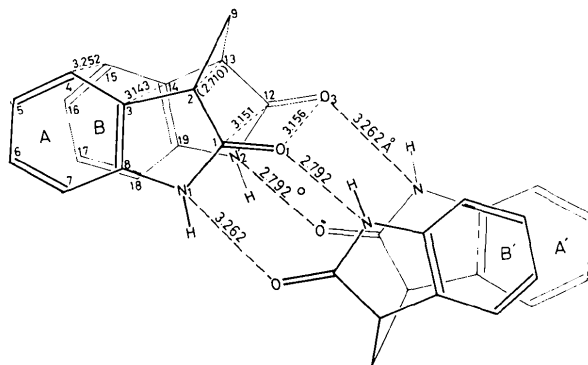


Fig. 5. Stacking pattern viewed perpendicular to the oxindole ring *A*. This also illustrates the dimeric structure formed by four hydrogen bonds.

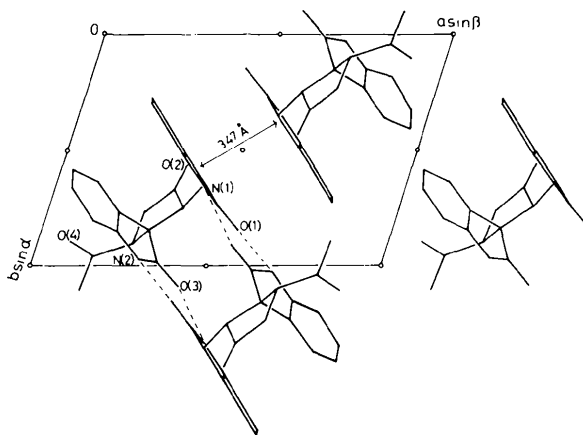


Fig. 6. A projection of the crystal structure viewed along the *c* axis.

A and *B* rings is 3.32 Å. The two molecules related by a center of symmetry at (0.5, 0, 0.5) are linked by four NH...O=C hydrogen bonds, forming a dimeric structure as illustrated in Fig. 5. Two of them, O(1)...N(2') and O(1')...N(2), are strong (2.792 Å) while the other two, O(3)...N(1') and O(3')...N(1), are weak (3.262 Å). The crystal structure viewed along the *c* axis is shown in Fig. 6. The two oxindole rings *A*, which are related by a center of symmetry at (0.5, 0.5, 0.5), are arranged nearly parallel to each other with a perpendicular distance of 3.47 Å, but they have little overlapping of atoms.

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