we follow the hydrogen bonds from one hydroxyl group to the next, we find chain-like structures of the type O—H···O—H···O—H··· which in some cases close up to form circles comprising five and six OH groups (Saenger, 1979). Similar endless chains have been observed previously in carbohydrate crystal structures (Jeffrey & Takagi, 1978; Jeffrey & Lewis, 1978; Jeffrey, Gress & Takagi, 1977). Theoretically, they have been explained on the basis of quantum-chemical calculations as being due to the 'cooperative effect' (Del Bene & Pople, 1973; Frank & Wen, 1957) which renders the hydroxyl group a stronger donor/acceptor if it already accepts/donates a hydrogen bond.

As reported for other crystal structures of a-CD (Saenger, 1976) the acetal oxygens O(4) and O(5) are not involved in hydrogen bonds. The recent X-ray analysis (Lindner & Saenger, to be published) of a second modification of $(\text{a-CD})_2.6H_2O$, however, demonstrated that in a-CD such interactions, at least to O(5), are indeed possible, in agreement with the rare observation of such hydrogen bonds in crystal structures of individual glucoses (Jeffrey & Takagi, 1978).

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The Structure of 4-(3-Indolyl)butyric Acid

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Abstract

$C_{12}H_{13}NO_2$ crystallizes in the monoclinic space group $P2_1/c$ with $a = 21.003$ (3), $b = 5.114$ (1), $c = 9.918$ (2) Å, $\beta = 93.278$ (6)°, $Z = 4$. The structure was solved by direct methods and refined to $R = 0.056$ for 1729 counter reflections. O—H···O bonds of length 2.644 Å are observed.

Introduction

The best established and most widely occurring natural auxin is indolylacetic acid (IAA). Homologues of IAA

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* Contribution No. 515.

such as 4-(3-indolyl)butyric acid (IBA) are also known to possess growth-regulating activity (Steward & Krikorian, 1971). The activity index, measured as a percentage of the molar concentration of indolylacetic acid inducing an elongation of 0·015 mm to the molar concentration of growth regulator inducing the same elongation, is 15 for IBA. Both IAA and IBA show a root-promoting effect in all lemon and lime varieties (Sircar, 1971) and IBA is also effective in bud inhibition in many plants.

The capacity of IBA to cause development of adventitious roots in profusion is so dependable that it is a widely used horticultural device (Galston & Davies, 1970). Cuttings of plants that do not normally root spontaneously may be dipped basally into a solution or powder of the auxin and then planted in a sand rooting bed. Extensive rooting follows within days to weeks, depending upon the plant.

There has been evidence implicating nucleic acids in plant growth (Thimann, 1969) and most of the observed phenomena can be interpreted on the basis that auxins function by somehow activating a messenger RNA, thereby inducing synthesis of specific enzymes. These would bring about insertion of new materials into the cell wall, causing its extension. In this context, the spatial similarity between the indole and purine rings is obviously suggestive.

Crystal data

4-(3-Indolyl)butyric acid (IBA), $C_{12}H_{13}NO_2$, $M_r = 203·2$, monoclinic, $a = 21·003$ (3), $b = 5·114$ (1), $c = 9·918$ (2) Å, $\beta = 93·278$ (6)°, $V = 1063$ Å^3, $Z = 4$, $D_m = 1·269$, $D_c = 1·270$ Mg m^{-3}, space group $P2_1/c$.

Experimental

IBA (Fig. 1) was crystallized from a mixture of benzene and petroleum ether. Preliminary cell parameters were obtained from Weissenberg photographs and precise values by least-squares refinement (Main & Woolfson, 1963) from 54 Bragg angles measured at
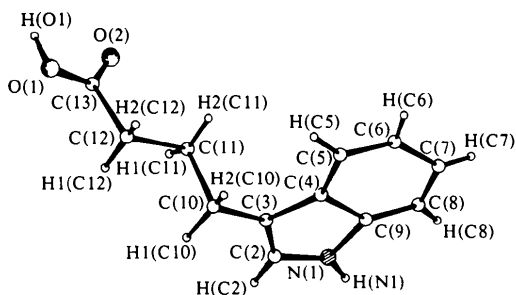
$\pm 2\theta$ (ranging from 25 to 92°) on a Picker four-circle diffractometer.

Intensities were collected with Ni-filtered Cu $K\alpha$ radiation ($\lambda = 1·5418$ Å) and a crystal $0·8 \times 0·4 \times 0·3$ mm mounted with **b** parallel to the φ axis of the diffractometer. Reflections within the range $2\theta \le 130°$ were scanned in the $\omega–2\theta$ mode at a scan rate of 2° min^{-1} and scan width of 2°. Backgrounds were measured at each extremum of the scan width for 10 s. A standard reflection monitored periodically showed no systematic drift. 1729 independent non-zero reflections were measured and corrected for Lorentz, polarization and background effects. The reflection 020 was used to record the intensity as a function of φ and absorption corrections were applied (North, Phillips & Mathews, 1966). The overall isotropic temperature and scale factors were estimated by a Wilson (1942) plot with the program of Hall (1968).

Structure determination and refinement

The structure was solved by direct methods (Karle & Karle, 1963, 1966). All but 16 of the 209 reflections with $|E| \ge 1·5$ were phased in terms of a single set of 203 signs. A subsequent E map revealed all the non-hydrogen atoms ($R = 0·437$).

Block-diagonal least-squares refinement, first with isotropic then anisotropic thermal parameters and unit weights for all reflections, reduced R to 0·099. For further refinement, weights $w = [\sigma(F)]^{-2}$ derived from counting statistics were introduced and yielded an R of 0·095. A difference synthesis then revealed all the H atoms close to their expected positions.

Full-matrix least-squares refinement with the program of Gantzel, Sparks & Trueblood (1961), with anisotropic thermal parameters for the non-hydrogen atoms and isotropic for H, led to convergence at $R = 0·056$.* The final shift in the atomic parameters of the non-hydrogen atoms was less than one-fifth the e.s.d. The residual electron density in a final difference map calculated with all the data did not exceed 0·2 e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1968).

Results and discussion

Final atomic parameters are listed in Tables 1 and 2. A view of the molecule along [001] is shown in Fig. 2. Interatomic distances and angles are listed in Tables 3 and 4.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35098 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 1. 4-(3-Indolyl)butyric acid: perspective view with atom numbering.

Table 1. *Fractional positional parameters* (×10⁴) *of the non-hydrogen atoms, with e.s.d.'s in parentheses*

	x	y	z
N(1)	3246 (1)	2305 (4)	10468 (2)
C(2)	2627 (1)	3142 (5)	10481 (2)
C(3)	2520 (1)	5128 (4)	9592 (2)
C(4)	3114 (1)	5552 (3)	8981 (2)
C(5)	3319 (1)	7313 (4)	8022 (2)
C(6)	3936 (1)	7233 (5)	7650 (2)
C(7)	4365 (1)	5434 (5)	8217 (3)
C(8)	4192 (1)	3677 (5)	9155 (3)
C(9)	3558 (1)	3748 (4)	9540 (2)
C(10)	1908 (1)	6555 (5)	9298 (2)
C(11)	1552 (1)	5684 (4)	7997 (2)
C(12)	938 (1)	7151 (4)	7705 (2)
C(13)	543 (1)	6197 (4)	6502 (2)
O(1)	43 (1)	7597 (3)	6183 (1)
O(2)	682 (1)	4224 (3)	5874 (1)

Table 2. *Fractional positional* (×10³) *and isotropic thermal parameters of the hydrogen atoms, with e.s.d.'s in parentheses*

	x	y	z	B (Å²)
H(N1)	345 (1)	106 (6)	1090 (3)	7·7*
H(C2)	235 (1)	232 (4)	1107 (2)	7·6
H(C5)	300 (1)	858 (5)	761 (2)	5·4
H(C6)	410 (1)	856 (5)	702 (2)	5·8
H(C7)	482 (1)	539 (5)	789 (2)	6·1
H(C8)	445 (1)	253 (5)	950 (3)	5·9
H1(C10)	165 (1)	610 (6)	1004 (3)	8·3
H2(C10)	201 (1)	847 (6)	909 (3)	7·6
H1(C11)	145 (1)	372 (5)	805 (2)	6·3
H2(C11)	184 (1)	598 (5)	720 (2)	4·9
H1(C12)	69 (1)	688 (4)	847 (2)	5·2
H2(C12)	102 (1)	889 (5)	749 (2)	5·2
H(O1)	−22 (1)	699 (5)	535 (3)	7·6

* The e.s.d.'s in B range from 0·5 to 0·7 Å², with an average value of 0·6 Å².

Table 3. *Bond lengths* (Å) *with e.s.d.'s in parentheses*

N(1)–C(2)	1·369 (3)	C(13)–O(2)	1·230 (2)
C(2)–C(3)	1·355 (3)	N(1)–H(N1)	0·87 (3)
C(3)–C(4)	1·435 (2)	C(2)–H(C2)	0·95 (2)
C(4)–C(5)	1·395 (3)	C(5)–H(C5)	1·00 (2)
C(5)–C(6)	1·369 (3)	C(6)–H(C6)	1·00 (2)
C(6)–C(7)	1·384 (3)	C(7)–H(C7)	1·01 (2)
C(7)–C(8)	1·358 (4)	C(8)–H(C8)	0·85 (2)
C(8)–C(9)	1·406 (3)	C(10)–H1(C10)	0·97 (3)
C(9)–N(1)	1·375 (3)	C(10)–H2(C10)	1·02 (3)
C(9)–C(4)	1·402 (3)	C(11)–H1(C11)	1·03 (2)
C(3)–C(10)	1·492 (3)	C(11)–H2(C11)	1·03 (2)
C(10)–C(11)	1·521 (3)	C(12)–H1(C12)	0·96 (2)
C(11)–C(12)	1·506 (3)	C(12)–H2(C12)	0·93 (2)
C(12)–C(13)	1·495 (2)	O(1)–H(O1)	1·01 (3)
C(13)–O(1)	1·295 (2)		

Table 4. *Bond angles* (°) *with e.s.d.'s in parentheses*

|			
N(1)–C(2)–C(3)	110·5 (2)	H(C2)–C(2)–C(3)	131 (1)
C(2)–C(3)–C(4)	105·8 (2)	C(4)–C(5)–H(C5)	118 (1)
C(3)–C(4)–C(9)	107·9 (2)	H(C5)–C(5)–C(6)	122 (1)
C(4)–C(9)–N(1)	106·7 (2)	C(5)–C(6)–H(C6)	121 (1)
C(9)–N(1)–C(2)	109·1 (2)	H(C6)–C(6)–C(7)	118 (1)
C(3)–C(4)–C(5)	134·0 (2)	C(6)–C(7)–H(C7)	121 (1)
C(4)–C(5)–C(6)	119·8 (2)	H(C7)–C(7)–C(8)	117 (1)
C(5)–C(6)–C(7)	120·9 (2)	C(7)–C(8)–H(C8)	123 (2)
C(6)–C(7)–C(8)	121·7 (2)	H(C8)–C(8)–C(9)	119 (2)
C(7)–C(8)–C(9)	117·6 (2)	C(3)–C(10)–H1(C10)	105 (2)
C(8)–C(9)–C(4)	121·8 (2)	C(3)–C(10)–H2(C10)	109 (2)
C(8)–C(9)–N(1)	131·5 (2)	H1(C10)–C(10)–H2(C10)	121 (2)
C(9)–C(4)–C(5)	118·1 (2)	H1(C10)–C(10)–C(11)	108 (2)
C(2)–C(3)–C(10)	127·1 (2)	H2(C10)–C(10)–C(11)	102 (2)
C(4)–C(3)–C(10)	127·1 (2)	C(10)–C(11)–H1(C11)	110 (1)
C(3)–C(10)–C(11)	113·5 (2)	C(10)–C(11)–H2(C11)	109 (1)
C(10)–C(11)–C(12)	113·1 (2)	H1(C11)–C(11)–H2(C11)	109 (2)
C(11)–C(12)–C(13)	114·9 (2)	H1(C11)–C(11)–C(12)	108 (1)
C(12)–C(13)–O(1)	114·8 (2)	H2(C11)–C(11)–C(12)	108 (1)
C(12)–C(13)–O(2)	122·3 (2)	C(11)–C(12)–H1(C12)	106 (1)
O(1)–C(13)–O(2)	122·9 (2)	C(11)–C(12)–H2(C12)	111 (1)
		H1(C12)–C(12)–H2(C12)	116 (2)
C(9)–N(1)–H(N1)	119 (2)	H1(C12)–C(12)–C(13)	106 (1)
H(N1)–N(1)–C(2)	132 (2)	H2(C12)–C(12)–C(13)	103 (1)
N(1)–C(2)–H(C2)	119 (1)	C(13)–O(1)–H(O1)	115 (2)
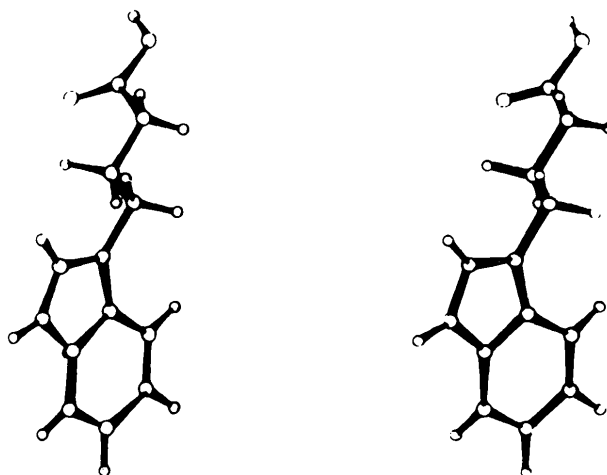
Fig. 2. Stereoscopic view down [001].

The six C—C lengths in the benzene ring average 1·386 (3) Å. In the five-membered ring, the two C—N bonds are of nearly equal length [averaging 1·372 (3) Å] and fall between the C=C [1·355 (3) Å] and C—C [1·435 (3) Å] lengths. Falkenberg & Carlström (1971) have tabulated the bond lengths in the indole ring in various structures and their data, coupled with information from more recent structural investigations, such as DL-tryptophan ethyl ester hydrochloride (Vijayalakshmi & Srinivasan, 1975), indole (Roychowdhury & Basak, 1975), and the present study, lead to the finding that in all indole derivatives the C(3)—C(4) bond is longer than the standard aromatic C—C bond of 1·395 Å.

The individual bond lengths in the indole ring differ appreciably from the values reported by Karle, Britts &

Table 5. *Equations of least-squares planes and distances (Å) of atoms from them, with e.s.d.'s in parentheses*

(i) Plane through N(1), C(2), C(3), C(4), C(5), C(6), C(7), C(8), C(9)

$$0.2249X + 0.6542Y + 0.7220Z = 9.6455$$

N(1)	−0.010 (2)	C(6)	−0.006 (2)
C(2)	−0.007 (2)	C(7)	−0.005 (3)
C(3)	0.003 (2)	C(8)	0.006 (2)
C(4)	0.009 (2)	C(9)	0.011 (2)
C(5)	−0.003 (2)		

(ii) Plane through C(3), C(10), C(11), C(12), C(13)

$$0.5186X + 0.6563Y − 0.5479Z = −1.0135$$

C(3)	−0.007 (2)	C(12)	0.028 (2)
C(10)	−0.026 (2)	C(13)	−0.033 (2)
C(11)	0.038 (2)		

(iii) Plane through C(12), C(13), O(1), O(2)

$$0.5740X + 0.5585Y − 0.5987Z = −1.6445$$

C(12)	−0.001 (2)	O(1)	−0.001 (1)
C(13)	0.003 (2)	O(2)	−0.001 (1)

Table 6. *Angle between the planes of the ring system and the carboxyl group in some plant hormones*

	Hormone		Reference
(1)	3-Indolylacetic acid (IAA)	90.0°	Karle, Britts & Gum (1964)
(2)	4-(3-Indolyl)butyric acid (IBA)	93.7	Present study
(3)	2-Chlorophenoxyacetic acid (2-CPA)	7.0; 6.6	Chandrasekhar & Pattabhi (1977)
(4)	2,4-Dichlorophenoxyacetic acid (2,4-D)	85.2	Smith, Kennard & White (1976)
(5)	1-Naphthaleneacetic acid (1-NAA)	98.7	Rajan (1978)
(6)	2-Naphthyloxyacetic acid (2-NAA)	4.2	Pattabhi, Raghunathan & Chacko (1978)
(7)	2-Coumaric acid (2-CA)	4.8	Raghunathan & Pattabhi (1979)
(8)	4-Coumaric acid (4-CA)	5.0	Utsumi, Fujii, Irie, Furusaki & Nitta (1970)
(9)	Kinetin	79.1	Soriano-Garcia & Parthasarathy (1977)
(10)	*trans*-β-2-Furylacrylic acid (β-2FA)	5.7	Filippakis & Schmidt (1967)

Fig. 3. Packing of the molecule viewed down b. Hydrogen bonds are shown by broken lines.

Templeton, 1962), but C(12)—C(13) is shorter by 0.045 Å. The two C—O bonds in the carboxyl group are of the same order of magnitude as in many other carboxylic acids (Nardelli, Fava & Giraldi, 1962).

The molecule of IBA is characterized by three planes, through the indole ring, the butyric group and the acid group. Details of these planes are given in Table 5. The plane of the indole ring makes angles of 43.8, 74.6 and 49.1° with the *ab*, *bc* and *ac* planes respectively. The planes containing butyric and acid groups are inclined at 7.1°, while the angle between the planes defined by the indole ring and the carboxyl group is 93.7°. The corresponding angles between the ring-system and carboxyl-group planes in a number of plant hormones are listed in Table 6 for comparison.

The packing of the IBA molecule is illustrated in Fig 3 in a view down **b**. The molecules exist as dimers. The carboxyl groups are hydrogen bonded across centres of symmetry and are therefore in parallel planes. The two carboxyl groups are, in fact, nearly coplanar, the planes being separated by only 0.093 Å. The O—H···O angle is 173.3° while the O···O distance of 2.644 Å is of the same order of magnitude as in many other acid dimers. There is no other intermolecular contact <3.4 Å.

Of the ten plant hormones listed in Table 6, five are essentially planar molecules (2-CPA, 2-NAA, 2-CA, 4-CA, β-2FA), while in the others the ring system is nearly perpendicular to the plane of the carboxyl group. All the structures are, as expected, stabilized by O—H···O-type hydrogen bonds of length ~2.65 Å, except in 2-CPA where the O—H···O bonds are much shorter (~2.47 Å). Further, the hydrogen-bonded carboxyl groups are nearly coplanar in each case. In both IAA and IBA, the N atom in the indole ring is not involved in hydrogen bonding. The bond lengths and angles in similar structures (taken in pairs starting from the first) generally agree within 2σ. A more detailed comparative study of the structural features of plant hormones will be reported elsewhere.

Gum (1964) for 3-indolylacetic acid (IAA), the maximum deviation being 0.051 Å for C(7)—C(8). However, the e.s.d.'s in the latter structure are about six times those for the present study, so that the bond lengths in the two compounds would agree generally within 2σ. The bond angles associated with the indole ring in IBA are roughly equal to the corresponding values in IAA.

Two of the C—C bonds in the butyric group, C(10)—C(11) [1.521 (3) Å] and C(11)—C(12) [1.506 (3) Å], are comparable in length to the corresponding bonds in butyric acid (Strieter &
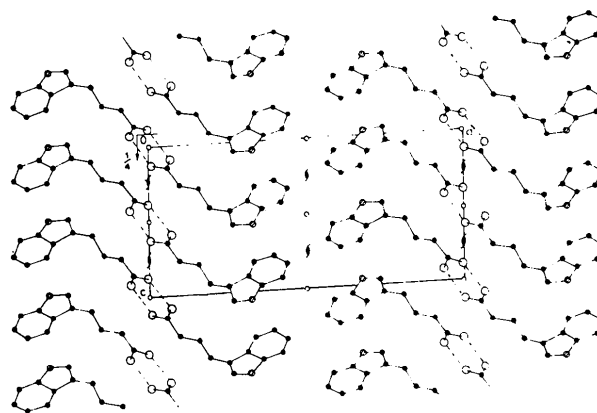
Possible hypotheses for the structural basis for the hormonal activity of auxins have been suggested by Kaethner (1977) and Thimann (1963). While Kaethner proposes a possibility of the bound auxin undergoing a simultaneous conformational change or reorientation with the receptor while in the binding site, Thimann postulates the existence of a fractional positive charge separated by 5·5 Å from a stronger negative charge arising from the dissociation of the carboxyl group as responsible for auxin activity. Indeed, in IAA the N atom in the indole ring is about 5·4 Å from the carboxyl group, but in IBA the corresponding distance is 8·1 Å. Perhaps the charge-separation theory could be coupled with the conformational-change hypothesis to yield a suitable explanation for auxin structure–activity relationships. The relative activities of various auxins probably depend on the position of the positive charge.

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