the preferred occurrence of only one form for two related molecules in three different crystal structures suggests that the conformation of tubercidin or its 2′-deoxy analogue is comparatively 'rigid'.

The crystal structures of tubercidin crystallized from water (Stroud, 1973; Abola & Sundaralingam, 1973) and of anhydrous 2′-deoxytubercidin (I) (Fig. 2) which was also crystallized from water are comparable, whereas the dihydrate (II) differs considerably. As indicated in Fig. 3, the crystal structure of the dihydrate consists of stacked bases along **c**, with a base–base separation of $c/2 = 3{\cdot}46$ Å, a classical value for bases of nucleic acids. The stacks are separated in the **a** direction by two water molecules and in the **b** direction by the sugar moieties. The water molecules, sugar residues and bases are involved in an extended hydrogen-bond network.* It is unfortunate that the water H atoms could not be located and therefore the hydrogen-bonding scheme is not defined with respect to the water molecules.

In the crystal structures of tubercidin and anhydrous 2′-deoxytubercidin the molecules are arranged such that bases and sugars form layers which are held together by hydrogen bonds. The bases are not stacked parallel to each other as in the dihydrate structure but adjacent bases are tilted in opposite sense about the C(4)–C(5) axis in tubercidin and approximately about the long axis C(8)–C(2) in 2′-deoxytubercidin.

* See deposition footnote.

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Acta Cryst. (1987). C**43**, 134–136

Structure of 1*H*-Indole-3-ethylenesalicylaldimine (sal TPA)*

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(*Received* 30 *April* 1986; *accepted* 24 *July* 1986)

Abstract. $C_{17}H_{16}N_2O$, $M_r = 264{\cdot}33$, orthorhombic, $Pca2_1$, $a = 15{\cdot}731$ (1), $b = 6{\cdot}035$ (1), $c = 14{\cdot}533$ (1) Å, $V = 1379{\cdot}7$ (2) Å^3, $Z = 4$, $D_m = 1{\cdot}23$, $D_x = 1{\cdot}27$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}599$ mm^{-1}, $F(000) = 560$, room temperature, final $R = 0{\cdot}037$ for 691 observed reflections. The present study established the molecular structure of the title compound. Structural features are compared with those of other similar compounds described as radiation protective agents. The C(1)–C(2) bond length of 1·36 (1) Å is significantly shorter than a normal aromatic C–C bond. There is an intramolecular hydrogen bond between the phenolic H and the N of the imino group.

* Systematic name: 2-[2-(1*H*-indol-3-yl)ethyl]iminomethylphenol.

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0108-2701/87/010134-03$01.50

Introduction. Tryptamine (1*H*-indole-3-ethanamine: a tryptophan metabolite with pharmacodynamic action) is able to condense aromatic carbonyl compounds. In this way with salicylaldehyde it forms the title compound (Soerens, Saudrin, Ungeniach, Mokry, Wu, Yamanaka, Hutchins, Dipierro & Cook, 1979). Sal TPA reacts with metal ions giving complexes with varied stereochemistries (Martin Reyes, Gili, Martin Zarza, Medina Ortega & Díaz González, 1986).

Experimental. Sal TPA was obtained using methods previously described (Soerens, Saudrin, Ungeniach, Mokry, Wu, Yamanaka, Hutchins, Dipierro & Cook, 1979) but with ethanol instead of benzene as solvent, giving a good yield without Pictet–Spengler cyclization of the tryptamine. The compound was recrystallized as yellow square prismatic crystals from hot ethanol. The density was determined by pycnometry.

The lattice constants (from 8 reflections, $15 < 2\theta < 30°$) and intensities were obtained from the measurements of a crystal measuring $0{\cdot}20 \times 0{\cdot}25 \times 0{\cdot}45$ mm, with a Siemens AED-4 four-circle computer-controlled diffractometer, graphite-monochromated Cu $K\alpha$ radiation, ω–θ scan mode. Of the 695 independent reflections with $3 < 2\theta < 100°$, 691 (99%) were considered observed with $I > 3\sigma(I)$; index range h 0,14; k 0,5; l 0,13. Two standard reflection measured every 30 min showed no significant intensity decay. The data were corrected for Lorentz and polarization effects but not for absorption ($\mu = 0{\cdot}599$ mm^{-1}). The structure was solved by direct methods (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and Fourier syntheses (Stewart, Kundell & Baldwin, 1970).

Refinement by full-matrix least-squares analysis, $\sum \Delta F^2$ minimized, with anisotropic temperature factors for non-H atoms. Most of the H atoms were found in a difference synthesis map and the remaining placed in calculated positions [*HSEARCH* program: Fayos & Martinez Ripoll (1980)] and included in subsequent refinements with fixed isotropic contribution. Final $R = 0{\cdot}037$, $wR = 0{\cdot}037$, unit weights. Scattering factors from *International Tables for X-ray Crystallography* (1974), max. electron density in final difference map $0{\cdot}12$ e Å^{-3}, max. Δ/σ (for non-H atoms) $= 0{\cdot}35$.

Discussion. Table 1 gives the final atomic parameters* following the numbering scheme of Fig. 1. Table 2 lists bond angles and bond lengths.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43280 (17 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* ($\times 10^4$) *with e.s.d.'s in parentheses and* B_{eq} *values* (Å^2)

$$B_{eq} = \tfrac{4}{3}\sum_i \sum_j \beta_{ij} \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	B_{eq}
O	5232 (3)	890 (8)	4625	68 (2)
N(1)	2704 (3)	−7716 (9)	1283 (16)	45 (2)
N(2)	4924 (4)	−2769 (9)	3742 (15)	45 (2)
C(1)	3424 (4)	−6880 (11)	1704 (16)	43 (3)
C(2)	3265 (4)	−4822 (10)	2054 (16)	35 (2)
C(3)	2386 (4)	−4322 (10)	1853 (15)	35 (2)
C(4)	1860 (4)	−2508 (10)	2023 (16)	41 (3)
C(5)	1040 (5)	−2546 (13)	1674 (16)	53 (3)
C(6)	745 (4)	−4382 (15)	1162 (15)	53 (3)
C(7)	1232 (4)	−6213 (13)	1009 (16)	47 (3)
C(8)	2068 (4)	−6173 (11)	1363 (16)	37 (2)
C(9)	3891 (4)	−3282 (11)	2526 (16)	41 (3)
C(10)	4352 (4)	−4384 (12)	3324 (16)	46 (3)
C(11)	5719 (5)	−3046 (10)	3648 (16)	39 (3)
C(12)	6326 (4)	−1443 (10)	4012 (16)	32 (2)
C(13)	7200 (4)	−1813 (12)	3880 (16)	42 (3)
C(14)	7784 (4)	−280 (14)	4199 (16)	51 (3)
C(15)	7512 (5)	1634 (11)	4639 (16)	52 (3)
C(16)	6659 (5)	2028 (11)	4787 (16)	49 (3)
C(17)	6062 (4)	489 (11)	4478 (15)	45 (3)

Table 2. *Interatomic lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

O–C(17)	1·34 (1)	C(5)–C(6)	1·41 (2)
O–H	1·01 (1)	C(6)–C(7)	1·36 (1)
N(1)–C(1)	1·38 (2)	C(7)–C(8)	1·41 (1)
N(1)–C(8)	1·37 (1)	C(9)–C(10)	1·52 (2)
N(2)–C(10)	1·46 (1)	C(11)–C(12)	1·46 (1)
N(2)–C(11)	1·27 (1)	C(12)–C(13)	1·40 (1)
C(1)–C(2)	1·36 (1)	C(12)–C(17)	1·41 (2)
C(2)–C(3)	1·45 (1)	C(13)–C(14)	1·39 (1)
C(2)–C(9)	1·52 (2)	C(14)–C(15)	1·39 (2)
C(3)–C(4)	1·39 (1)	C(15)–C(16)	1·38 (1)
C(3)–C(8)	1·42 (2)	C(16)–C(17)	1·39 (1)
C(4)–C(5)	1·38 (1)		

C(17)–O–H	118·1 (5)	N(1)–C(8)–C(7)	129·5 (7)
C(1)–N(1)–C(8)	108·2 (6)	N(1)–C(8)–C(3)	108·7 (6)
C(10)–N(2)–C(11)	118·4 (6)	C(2)–C(9)–C(10)	112·7 (6)
N(1)–C(1)–C(2)	110·3 (5)	N(2)–C(10)–C(9)	108·6 (6)
C(1)–C(2)–C(9)	127·4 (6)	N(2)–C(11)–C(12)	121·3 (7)
C(1)–C(2)–C(3)	106·8 (6)	C(11)–C(12)–C(17)	122·0 (6)
C(3)–C(2)–C(9)	125·7 (6)	C(11)–C(12)–C(13)	119·0 (6)
C(2)–C(3)–C(8)	105·9 (5)	C(13)–C(12)–C(17)	119·0 (6)
C(2)–C(3)–C(4)	134·1 (6)	C(12)–C(13)–C(14)	120·0 (7)
C(4)–C(3)–C(8)	119·9 (6)	C(13)–C(14)–C(15)	120·0 (7)
C(3)–C(4)–C(5)	118·3 (6)	C(14)–C(15)–C(16)	121·2 (7)
C(4)–C(5)–C(6)	120·7 (6)	C(15)–C(16)–C(17)	119·3 (7)
C(5)–C(6)–C(7)	122·5 (7)	C(12)–C(17)–C(16)	120·4 (7)
C(6)–C(7)–C(8)	116·8 (7)	O–C(17)–C(16)	118·9 (6)
C(3)–C(8)–C(7)	121·7 (6)	O–C(17)–C(12)	120·7 (6)

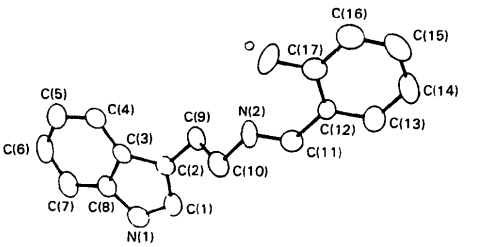

Fig. 1. View of the molecule drawn with *ORTEP* (Johnson, 1965).

The C(1)–C(2) bond of 1·36(1) Å is apparently shorter than a normal aromatic C–C bond (1·395 Å).

This has previously been described in tryptamine hydrochloride, a radiation protector (Wakahara, Fujiwara & Tomita, 1973) and tryptamine picrate (Gartland, Freeman & Bugg, 1974). However, in another derivative of tryptophan, the potent mutagenic 3-amino-1,4-dimethyl-5*H*-pyrido[4,3-*b*]indole (Itai & Iitaka, 1978), the C(1)–C(2) bond is 1·428 (4) Å. Most torsion angles (Klyne & Prelog, 1960) are close to 0 or 180°. The non-hydrogen atoms lie in two different planes connected through the C(9)–C(10) single bond.

The C(2)–C(9)–C(10)–N(2) torsion angle (179°) indicates an antiperiplanar relationship across the C(9)–C(10) bond. Other significant torsion angles are −54° (synclinal), 129° (anticlinal) and −111° (anticlinal) for C(1)–C(2)–C(9)–C(10), C(3)–C(2)–C(9)–C(10) and C(11)–N(2)–C(10)–C(9) respectively.

An intramolecular hydrogen bond is found between the N(2) atom and the oxygen atom of the phenol ring. The N···H–O distance is 2·60 (2) Å and the angle is 131·9 (3)°.

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Acta Cryst. (1987). C**43**, 136–139

Structure of the Acetone Solvate of 17α-Hydroxy-3,11,20-trioxo-4-pregnen-21-yl Acetate (Cortisone Acetate, Modification IV*ac*)

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(*Received* 17 *June* 1986; *accepted* 18 *July* 1986)

Abstract. $C_{23}H_{30}O_6.C_3H_6O$, M_r=460·57, monoclinic, $P2_1$, a = 9·820 (2), b = 7·661 (5), c = 16·648 (1) Å, β = 94·65 (1)°, V = 1248·3 (9) Å^3, Z = 2, D_x = 1·225 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ (Mo $K\alpha$) = 0·8 cm^{-1}, $F(000)$ = 496, room temperature, R = 0·063 for 1587 unique reflections with $I \geq 2{\cdot}5\sigma(I)$. The crystal structure of the acetone solvate, which is rather unstable, is isomorphous with the ethanol solvate. The conformation of the steroid molecule is identical to that of anhydrous modification II and deviates from that of anhydrous modification I by the conformation of ring *D* and the side-chain orientation with respect to the steroid skeleton. The acetone solvate is disordered over two positions (2:1) and hydrogen bonded with O(17) of the steroid molecule.

Introduction. In the crystalline state three anhydrous polymorphs of cortisone acetate (CA) exist (*e.g.* Callow & Kennard, 1961) and numerous pseudo-polymorphic forms (solvates) have been reported (Callow & Kennard, 1961; Carless, Moustafa & Rapson, 1966; Kuhnert-Brandstätter & Grimm, 1968; Shirotani & Sekiguchi, 1981). Structure analyses of anhydrous modifications I (CA I) and II (CA II) have been reported by Kanters, de Koster, van Geerestein & van Dijck (1985) and Declercq, Germain & Van Meerssche (1972), respectively. In the literature there is confusion about the correct description and designation of the different forms of CA because of possible interconversions (Mesley, 1968; van Geerestein, Kanters, van Dijck & van Wendel de Joode, 1985). The nomenclature of Carless, Moustafa & Rapson (1966) will be followed here. This paper reports the analysis of the acetone solvate of CA and is intended to be the first paper in a series on the X-ray structures of solvates of CA. The monoacetonate has not yet been described in

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