Bei den bekannten Strukturen mit Sb–F–Sb-Brücken wird dagegen durchweg eine Abstandsvergrösserung $Sb–F_{Brücke} > Sb–F_{terminal}$ und eine Aufweitung des Winkels Sb–F–Sb ∼ 150° oder grösser gefunden, z. B. $(Sb_2F_{11})^-$ $[(XeF)^+(Sb_2F_{11})^-$: McRae, Peacock & Russell (1969); $(SbCl_4)^+(Sb_2F_{11})^-$: Miller, Baird, Bramlett & Templeton (1972); $(BrF_4)^+(Sb_2F_{11})^-$: Lind & Christe (1971)], $(Sb_3F_{16})^-$ $[(Br_2)^+(Sb_3F_{16})^-$: Edwards & Jones (1971)] und $Sb_{11}F_{43}$: Edwards & Slim (1974). Im Unterschied zur Sb–O–Sb-Brücke des $(Sb_2F_{10}O)^{2-}$ Anions sollte die Sb–F–Sb-Brücke weniger stabilisiert sein.

Das Gesamtmittel der Sb–F-Abstände beträgt 1,88(2) Å (Tabelle 2), vergleichsweise bei $Cs_2(Sb_2F_{10}O)$ 1,872(13) Å und bei $Cs_3(Sb_3F_{12}O_3)$ 1,88(2) Å.

Im Gegensatz zum Anion in der Verbindung $Cs_2(Sb_2F_{10}O)$ mit $2(C_2)$-Symmetrie hat das Anion im $Rb_2(Sb_2F_{10}O)$ nur die triviale Symmetrie $1(C_1)$. Die Symmetrie $m(C_s)$, die für das Anion $(As_2F_{10}O)^{2-}$ durchgängig nachgewiesen wurde, wird bei $(Sb_2F_{10}O)^{2-}$ nicht verifiziert. Infolge der gegenüber den Abständen As–F,O grösseren Abstände Sb–F,O wird im Anion $(Sb_2F_{10}O)^{2-}$ demzufolge nicht notwendigerweise die 'dichteste' Packung $m(C_s)$ (Haase, 1973*b*) eingenommen.

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The Orthorhombic Form of *p*-Hydroxyacetanilide

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Abstract. $HO–C_6H_4NHCOCH_3$, m.p. 160–161 °C, orthorhombic, *Pcab*, $a = 11{\cdot}805(5)$, $b = 17{\cdot}164(2)$, $c = 7{\cdot}393(2)$ Å. $\varrho_{obs} = 1{\cdot}34$, $\varrho_{calc} = 1{\cdot}342$ g cm^{-3}, $Z = 8$. The structure was refined to $R = 0{\cdot}077$ for 1098 non-zero reflexions. The two kinds of hydrogen bonds [2·724(5) Å, OH donates to O=C; 2·967(5) Å, OH accepts from H–N] form two-dimensional molecular sheets parallel to the *ab* plane and the sheets are stacked along the *c* axis. The carbonyl group takes *endo* conformation to the benzene ring as usual and the dihedral angle between the benzene ring and amide planes is 17·7°.

Introduction. The orthorhombic crystals obtained by slow evaporation from an ethanol solution were prisms elongated along the *c* axis. Systematic absences were *h*0*l* when *h* odd, *hk*0 when *k* odd, 0*kl* when *l* odd. Lattice constants and intensities were measured from a crystal of dimensions 0·21 × 0·19 × 0·21 mm, on an automatic four-circle diffractometer with graphite-monochromatized Mo *Kα* radiation and a scintillation counter with pulse-height analyser. The $\omega/2\theta$-scan method was employed (scan speed in 2θ: 4° min^{-1}; scan range in 2θ: $1{\cdot}3 + 0{\cdot}35 \tan \theta_B$, where θ_B is Bragg angle). Background was measured for 10 s on either side of the peak. 1714 independent reflexions were surveyed up to $2\theta = 55°$, and 1098 non-zero reflexions were obtained. The corrections for Lorentz and polarization factors were applied, but no absorption correction was made [μ(Mo $K\alpha$) = 1·05 cm^{-1}].

Taking into consideration intense reflexions, 1,14,0, 10,4,0 and 380, the trial structure was obtained from a sharpened Patterson map. Diagonal least-

Table 1. *The final atomic coordinates and thermal parameters* ($\times 10^4$) *with standard deviations in parentheses*

The anisotropic coefficients have the form exp $(-\beta_{11}h^2-\beta_{22}k^2-\beta_{33}l^2-\beta_{12}hk-\beta_{13}hl-\beta_{23}kl)$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O(1)	2630 (2)	1324 (1)	2688 (5)	47 (2)	21 (1)	320 (8)	−8 (2)	−13 (7)	5 (4)
O(2)	3969 (2)	5030 (2)	2288 (5)	54 (2)	21 (1)	435 (10)	8 (2)	−58 (8)	9 (5)
N	5181 (2)	4053 (2)	2912 (5)	35 (2)	20 (1)	236 (8)	−1 (2)	−24 (7)	−2 (5)
C(1)	4462 (3)	3387 (2)	2910 (5)	34 (2)	20 (1)	153 (7)	−1 (2)	3 (7)	−11 (5)
C(2)	3391 (3)	3373 (2)	2122 (5)	40 (2)	19 (1)	178 (8)	13 (3)	−2 (7)	−3 (5)
C(3)	2791 (3)	2680 (2)	2073 (5)	39 (2)	24 (1)	146 (7)	0 (3)	−27 (7)	−17 (5)
C(4)	3241 (3)	2008 (2)	2807 (5)	37 (2)	19 (1)	160 (7)	1 (3)	26 (7)	−7 (5)
C(5)	4281 (3)	2026 (2)	3655 (5)	45 (2)	20 (1)	163 (8)	10 (3)	1 (7)	15 (5)
C(6)	4894 (3)	2718 (2)	3700 (5)	36 (2)	23 (1)	155 (7)	9 (3)	−16 (7)	−3 (5)
C(7)	4930 (3)	4805 (2)	2615 (6)	48 (2)	22 (1)	217 (9)	−1 (3)	0 (9)	−15 (6)
C(8)	5933 (3)	5350 (2)	2694 (7)	56 (3)	24 (1)	332 (13)	−17 (3)	−38 (11)	3 (7)

squares refinement (program *RSDLS*-3, Sakurai, 1967) reduced the R value from 0·418 to 0·185. A difference synthesis at this stage revealed all the hydrogen atoms. Block-diagonal least-squares refinement (*HBLS* V, Ashida, 1973) including all the atoms resulted in a final R of 0·077 for 1098 non-zero reflexions.* The weights adopted in the later stages of the refinement were $w=1{\cdot}0$ for $0<|F_o|\le 32{\cdot}0$, $w=1{\cdot}0/\{1{\cdot}0+0{\cdot}161(|F_o|-32{\cdot}0)\}$ for $|F_o|>32{\cdot}0$; the reflexions 002, 601 and 621 being excluded. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The computations were performed on a NEAC 2200-700 computer of the Computation Centre of Osaka University, and a NEAC 2200-500 computer of the Okayama University Computer Centre.

The final atomic parameters are listed in Tables 1 and 2. Bond lengths and angles are shown in Fig. 1, and the projections of the crystal structure in Fig. 2.

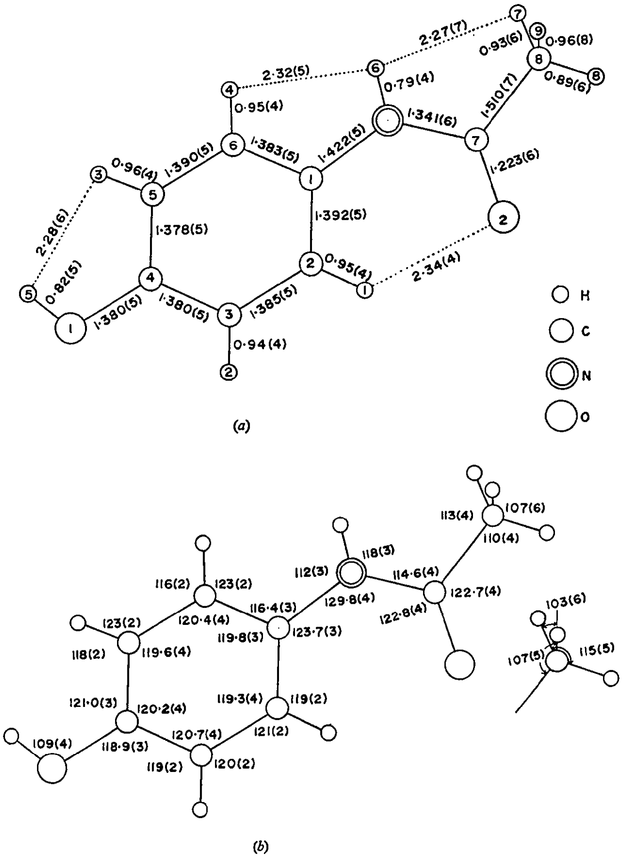

Fig. 1. The molecular structure; (*a*) bond lengths (Å) and numbering of atoms, (*b*) bond angles (°). Their estimated standard deviations are shown in parentheses.

Table 2. *The final parameters of the hydrogen atoms*

	x	y	z	B(Å^2)
H(1)	0·309 (3)	0·384 (2)	0·163 (5)	1·1 (0·8)
H(2)	0·207 (3)	0·266 (2)	0·154 (5)	1·3 (0·8)
H(3)	0·454 (3)	0·155 (2)	0·420 (5)	0·7 (0·7)
H(4)	0·559 (3)	0·271 (2)	0·433 (5)	0·5 (0·7)
H(5)	0·306 (4)	0·095 (3)	0·279 (7)	3·7 (1·2)
H(6)	0·582 (3)	0·395 (2)	0·314 (5)	0·7 (0·8)
H(7)	0·650 (5)	0·518 (3)	0·346 (8)	5·6 (1·5)
H(8)	0·571 (5)	0·583 (3)	0·289 (7)	5·2 (1·5)
H(9)	0·629 (7)	0·534 (5)	0·152 (11)	10·6 (2·4)

Discussion. Many derivatives of acetanilide have antipyretic and analgesic actions but show some undesirable side effects on the human body. It is of particular interest that *p*-hydroxyacetanilide is a metabolite of a carcinogen, 4-dimethylaminoazobenzene (Stevenson, Dobriner & Rhoads, 1942). We have obtained the orthorhombic form in addition to the monoclinic form (Groth, 1919), and have undertaken the structure analysis to examine the hydrogen-bond scheme and the degree of planarity of the molecule.

In the crystals, the *p*-hydroxyl group donates one hydrogen bond to the carbonyl O(2) of the molecule related by the b glide plane [O(1)–H(5)···O(2) = 171 (5)°, H(5)···O(2) = 1·91 (5) Å, O(1)···O(2) = 2·724 (5) Å] and accepts another hydrogen bond from the amide N of the molecule related by the a glide plane [O(1)···H(6)–N = 159 (4)°, O(1)···H(6) = 2·21 (4) Å, O(1)···N = 2·967(5) Å]. The two kinds of hydrogen bond form two-dimensional molecular sheets parallel to the ab plane and the sheets are stacked along the c axis with van der Waals contacts. The O(1)···H(6)–N

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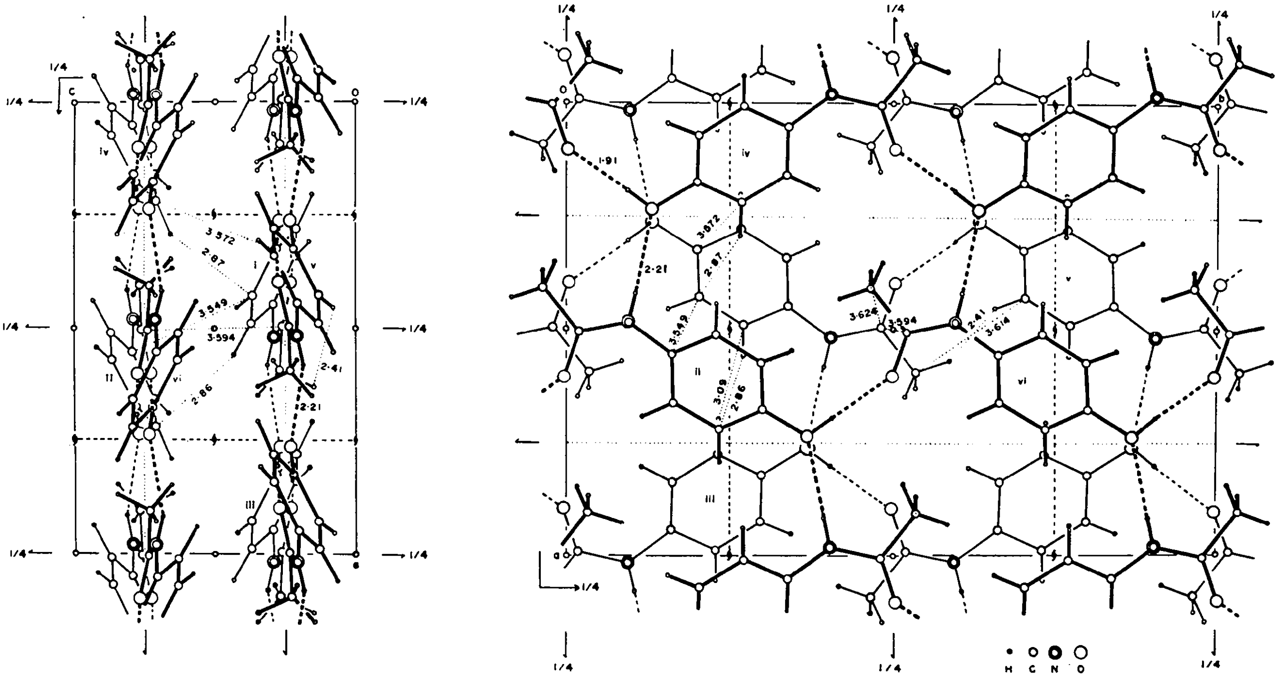
* A table of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30521 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.



(a) (b)

Fig. 2. The projections of the crystal structure; (*a*) viewed along the *b* axis, (*b*) viewed along the *c* axis. Broken lines show hydrogen bonds, and dotted lines intermolecular contacts. Symmetry code: (i) x,y,z; (ii) $1-x,\frac{1}{2}-y,\frac{1}{2}+z$; (iii) $\frac{1}{2}+x,\frac{1}{2}-y,z$; (iv) $\frac{1}{2}-x,y,\frac{1}{2}+z$; (v) $x,\frac{1}{2}+y,\frac{1}{2}-z$; (vi) $1-x,1-y,1-z$.

angle is rather small. The linearity of the hydrogen bond would be attained by taking a dihedral angle of about 30° between the amide group and the benzene-ring plane; the consequent lengthening of the shorter O(1)···O(2) hydrogen bond would presumably lead to less efficient packing.

The carbonyl group is in the *endo* conformation with respect to the benzene ring, as is usually observed. The benzene ring and acetamide group are planar within 0·018 and 0·004 Å, respectively. The dihedral angle between the planes is 17·7°, a value very close to the 17·6° found in acetanilide (Brown, 1966), in spite of the difference in the hydrogen-bond scheme, but much smaller than the 50–60° obtained from CNDO/2 calculations for acetanilide (Olsen & Kang, 1970). The smaller dihedral angles found in the crystals may be preferable for the molecular packing.

The bond lengths C(1)–N, N–C(7) and C(7)–O(2) agree well with those found in many acetanilides in spite of their various dihedral angles. The atoms N and O(1) deviate from the benzene ring plane by +0·111 and +0·060 Å, respectively. The deviations of the N atoms are 0·046 Å in acetanilide and 0·150 Å in *N*-methylacetanilide (Pedersen, 1967).

The C(1)–N–C(7) angle is significantly larger than the corresponding angle, 123°, in polypeptides (Corey & Pauling, 1953). Widening of this and the C(2)–C(1)–N angles and narrowing of the C(6)–C(1)–N angle seem to result from the intramolecular repulsion between O(2) and H(1); the distance O(2)···H(1) is significantly smaller than the sum of the van der Waals radii.

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