(1985) in the crystal structure of S,S-ethambutol dihydrochloride (SSET) (Table 3).

The structures of (SSET) and (RSET–295) are closely related. Although (SSET) crystallizes in the orthorhombic space group $P2_12_12$ ($Z = 2$), the cell constants are, however, nearly the same [$a = 23\cdot183$ (22), $b = 6\cdot555$ (5), $c = 5\cdot176$ (5) Å for (SSET)]. The differences between corresponding bond lengths and bond angles range from $0\cdot001$ to $0\cdot027$ Å and $0\cdot6$ to $1\cdot2°$, respectively.

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Structure of Ethyl 1,2-Dihydro-2-oxo-3,6-diphenyl-4-pyridinecarboxylate

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Abstract. $C_{20}H_{17}NO_3$, $M_r = 319\cdot3$, monoclinic, $P2_1/n$, $a = 9\cdot0385$ (8), $b = 9\cdot613$ (2), $c = 18\cdot879$ (3) Å, $\beta = 92\cdot895$ (9)°, $V = 1640$ (2) Å^3, $Z = 4$, $D_m = 1\cdot301$ (3) g cm^{-3}, $D_x = 1\cdot292$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot5418$ Å, $\mu = 6\cdot22$ cm^{-1}, $F(000) = 672$, $T = 295$ K, final $R(F) = 0\cdot0643$, $wR = 0\cdot0663$ for 1852 observed reflections, [$I \geq 2\cdot5\sigma(I)$]. In the unit cell the molecules are stabilized by N—H$\cdots$O and C—H$\cdots$O hydrogen bonds. Centrosymmetrically related molecules form dimers about the centre of inversion, and the hydrogen-bond pattern is reminiscent of pyrimidine-purine base pairing in nucleotides.

Introduction. Pyridine is generally used in the synthesis of vitamins and drugs and pyridine derivatives like isonicotinyl hydrazide are used in the treatment of tuberculosis (Metzler, 1977). The title compound is a metabolite of the anti-tubercular drug ethambutol [(R)-2,2'-(1,2-ethanediyldiimino)bis-1-butanol].   The structure solution was undertaken to verify the chemical structure.

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Experimental. Colourless, transparent crystals, $0\cdot4 \times 0\cdot3 \times 0\cdot25$ mm, rectangular, prismatic, were crystallized from methanol/water. Density measured by flotation. Three-dimensional intensity data using Enraf–Nonius CAD-4 automated diffractometer; $\omega/2\theta$ scan mode with max. $2\theta = 130°$, graphite-monochromated Cu $K\alpha$ radiation. Intensity data were corrected for polarization and Lorentz effects, not for absorption ($\mu t < 1$). Unit-cell parameters were refined from 25 reflections in the range $30 \leq 2\theta \leq 58°$. The intensities of 2968 unique reflections were measured within the limits $-10 \leq h \leq 10$, $0 \leq k \leq 11$, $0 \leq l \leq 28$; 1852 observed with $I \geq 2\cdot5\sigma(I)$; three standard reflections monitored every 100 reflections showed no significant variation. Structure solution by direct methods using *SHELXS*86 (Sheldrick, 1986). The structure was refined using full-matrix least-squares technique. The function minimized is $\sum w(|F_o| - |F_c|)^2$ where $w = 1/[\sigma^2(|F_o|) + 0\cdot036066 |F_o^2|]$ in *SHELX*76 (Sheldrick, 1976); H-atom positions from $\Delta\rho$ syntheses. Atoms C(23) and C(24) are disordered; two peaks of height $1\cdot5$ e Å^{-3} and $0\cdot6$ e Å^{-3} were located in the $\Delta\rho$ map at a distance of $0\cdot7$ Å to C(23) and $0\cdot6$ Å to C(24), respectively, and they have been included in the

Table 1. *Positional parameters* $(\times 10^4)$ *for non-H atoms with e.s.d.'s in parentheses*

	x	y	z	Occupancy	U_{eq}(Å²)*
N(1)	1004 (4)	3367 (3)	5382 (2)	1·0	0·042 (2)
C(2)	911 (4)	3185 (3)	4657 (2)	1·0	0·042 (2)
C(3)	1581 (4)	1941 (3)	4391 (2)	1·0	0·037 (2)
C(4)	2283 (4)	1058 (4)	4866 (2)	1·0	0·041 (2)
C(5)	2297 (4)	1293 (4)	5601 (2)	1·0	0·042 (2)
C(6)	1656 (4)	2462 (3)	5859 (2)	1·0	0·036 (2)
C(7)	1581 (4)	2770 (3)	6623 (2)	1·0	0·038 (2)
C(8)	2650 (5)	2214 (4)	7104 (2)	1·0	0·049 (2)
C(9)	2564 (5)	2445 (5)	7826 (2)	1·0	0·057 (3)
C(10)	1430 (6)	3278 (4)	8074 (2)	1·0	0·056 (3)
C(11)	379 (5)	3820 (4)	7601 (2)	1·0	0·053 (3)
C(12)	453 (5)	3574 (4)	6884 (2)	1·0	0·040 (2)
C(13)	1403 (4)	1660 (3)	3613 (2)	1·0	0·036 (2)
C(14)	1765 (5)	2635 (4)	3114 (2)	1·0	0·045 (2)
C(15)	1586 (5)	2345 (4)	2395 (2)	1·0	0·050 (2)
C(16)	1041 (5)	1075 (5)	2178 (2)	1·0	0·052 (2)
C(17)	651 (5)	100 (4)	2668 (2)	1·0	0·051 (2)
C(18)	839 (5)	394 (4)	3381 (2)	1·0	0·046 (2)
O(19)	279 (4)	4080 (3)	4275 (1)	1·0	0·060 (2)
C(20)	3149 (5)	−161 (4)	4614 (2)	1·0	0·046 (2)
O(21)	4094 (4)	−79 (3)	4195 (2)	1·0	0·068 (2)
O(22)	2769 (3)	−1320 (2)	4931 (1)	1·0	0·068 (2)
C(23)	3867 (8)	−2499 (7)	4797 (3)	0·588 (2)	0·053 (2)
C(23')	3147 (11)	−2693 (10)	4637 (5)	0·423 (5)	0·057 (2)
C(24)	3410 (13)	−3630 (11)	5244 (6)	0·719 (2)	0·121 (4)
C(24')	3016 (16)	−3766 (15)	4959 (8)	0·273 (3)	0·050 (3)

* $U_{eq} = \frac{1}{3}[U_{22} + 1/\sin^2\beta \, (U_{11} + U_{33} + 2U_{13}\cos\beta)]$.

refinement. The site occupancy factors and the iso-tropic thermal parameters of the disordered atoms were refined alternately. The H atoms attached to these carbons could not be located. All other H atoms were refined isotropically and all non-H atoms aniso-tropically. Final $R(F) = 0.0643$ and $wR = 0.0663$. $(\Delta/\sigma) \le 0.36$, with $(\Delta/\sigma)_{mean} = 0.06$ and $S = 0.52$ for 215 parameters. Final $(\Delta\rho)_{max}/(\Delta\rho)_{min} = 0.46/-0.43$ e Å⁻³. Scattering factors are as in *SHELX76* (Sheldrick, 1976).

Discussion. Atomic positions and equivalent tem-perature factors for non-H atoms are given in Table 1.* Fig. 1 shows a stereoview of the molecule. The bond lengths and angles involving the non-H atoms are given in Table 2. Fig. 2 shows the thermal ellipsoids drawn at the 50% probability level (Johnson, 1965). The average e.s.d.'s in bond lengths and angles are 0·006 Å and 0·03° respectively. The average C—C distance of 1·388 (6) Å and angle of 120·0 (3)° around the phenyl ring *B* (see Fig. 2), and 1·380 (6) Å and 120·0 (3)° around the phenyl ring *C*, agree with the standard values. Bond lengths and angles observed in this structure are comparable with the values observed in other related structures like 3-ethyl-6-phenyl-4-

Table 2. *Bond lengths* (Å) *and bond angles* (°) *involving non-H atoms with e.s.d.'s in parentheses*

N(1)—C(2)	1·378 (5)	C(7)—C(12)	1·390 (6)
N(1)—C(6)	1·365 (5)	C(13)—C(14)	1·381 (5)
C(2)—C(3)	1·444 (5)	C(14)—C(15)	1·387 (5)
C(2)—O(19)	1·244 (5)	C(15)—C(16)	1·373 (6)
C(3)—C(4)	1·368 (5)	C(16)—C(17)	1·377 (6)
C(3)—C(13)	1·494 (5)	C(17)—C(18)	1·378 (5)
C(4)—C(5)	1·405 (5)	C(18)—C(13)	1·384 (5)
C(4)—C(20)	1·502 (6)	C(20)—O(21)	1·196 (6)
C(5)—C(6)	1·367 (5)	C(20)—O(22)	1·320 (5)
C(6)—C(7)	1·477 (5)	O(22)—C(23)	1·537 (7)
C(7)—C(8)	1·399 (5)	C(23)—C(24)	1·45 (1)
C(8)—C(9)	1·387 (5)	O(22)—C(23')	1·48 (1)
C(9)—C(10)	1·401 (7)	C(23')—C(24')	1·47 (1)
C(10)—C(11)	1·374 (6)	C(23')—C(24')	1·21 (2)
C(11)—C(12)	1·379 (5)		

C(2)—N(1)—C(6)	125·2 (3)	C(8)—C(9)—C(10)	119·9 (3)
N(1)—C(2)—C(3)	116·4 (3)	C(9)—C(10)—C(11)	119·6 (3)
N(1)—C(2)—O(19)	119·4 (3)	C(10)—C(11)—C(12)	120·4 (3)
C(3)—C(2)—O(19)	124·2 (3)	C(11)—C(12)—C(7)	121·1 (3)
C(2)—C(3)—C(4)	118·5 (3)	C(3)—C(13)—C(14)	122·0 (3)
C(2)—C(3)—C(13)	117·8 (3)	C(3)—C(13)—C(18)	119·4 (3)
C(4)—C(3)—C(13)	123·7 (3)	C(14)—C(13)—C(18)	118·6 (3)
C(3)—C(4)—C(5)	121·9 (3)	C(13)—C(14)—C(15)	120·7 (3)
C(3)—C(4)—C(20)	120·7 (3)	C(14)—C(15)—C(16)	119·6 (3)
C(5)—C(4)—C(20)	117·4 (3)	C(15)—C(16)—C(17)	120·5 (3)
C(4)—C(5)—C(6)	120·1 (6)	C(16)—C(17)—C(18)	119·6 (3)
N(1)—C(6)—C(5)	117·9 (3)	C(17)—C(18)—C(13)	121·0 (3)
N(1)—C(6)—C(7)	118·4 (3)	C(4)—C(20)—O(22)	111·5 (3)
C(5)—C(6)—C(7)	123·7 (3)	C(4)—C(20)—O(21)	124·0 (3)
C(6)—C(7)—C(8)	119·5 (3)	O(21)—C(20)—O(22)	124·4 (3)
C(6)—C(7)—C(12)	121·9 (3)	C(20)—O(22)—C(23)	111·5 (3)
C(8)—C(7)—C(12)	118·6 (3)	O(22)—C(23)—C(24)	104·7 (5)
C(7)—C(8)—C(9)	120·4 (3)	O(22)—C(23')—C(24')	103·2 (6)

methoxycarbonyl-1*H*-2-pyridone (Nethaji, Vasantha Pattabhi & Gabe, 1989*a*), 3-methoxycarbonyl-2-pro-panoyl-5-phenyl-1*H*-pyrrole (Nethaji, Vasantha Pat-tabhi & Thailambal, 1989*b*) and 5,5'-dichloro-3,3'-dinitro-2,2'-biphenyldiol (Hay & Mackay, 1981). Table 3 gives the pyridine geometry observed in various structures where the N atom is protonated. It can be seen from the table that the geometry observed in the present study agrees well with the related structures.

The C(2)—O(19) bond is elongated [1·244 (4) Å] from a normal C=O bond distance and the actual bond distance is reported to be dependent on the hydrogen-bond interaction of this atom (Kvick & Booles, 1972). The stronger the hydrogen bond, the larger is the distance (Table 4). In this structure the N atom is protonated as in the case of 3-ethyl-6-phenyl-4-methoxy-carbonyl-1*H*-2-pyridone (Nethaji, Vasantha Pattabhi & Gabe, 1989*a*). The H atom covalently attached to N has been located from the $\Delta\rho$ map and its presence is further substantiated by the widening of the angle C(2)—N(1)—C(6), 125·2 (3)°, which is reported to be of the order of 123·5° in the protonated structures (Shieh & Voet, 1975). Angles C(4)—C(3)—C(13), 123·7 (3)°, and C(4)—C(20)—O(21), 124·0 (3)°, are also widened due to the steric repulsion between the bulky substituents (Brown, 1966). The widening of the angle C(5)—C(6)—C(7), 123·7 (3)° and the rotation of the phenyl ring C about C(6)—C(7) by 25·46° with

respect to the pyridine ring plane minimize the steric interaction between the H atoms HC(5) and HC(8) and between HN(1) and HC(12). It has been reported that the aromatic groups in these systems rotate about the exocyclic bond until the 2,2' atoms are separated by the sum of their van der Waals radii (Branch, Byron, Gray, Ibbotson & Worral, 1964). This angle of rotation varies between 25 and 90° depending on the substituent. However, the pyridine ring is rotated about the C(3)—C(13) bond by 51·6° minimizing the steric interaction between the multiple substituents and the H atoms of the phenyl group.

The phenyl rings B and C are planar while the pyridine ring is non-planar with $\chi^2 = 8\cdot16$, $16\cdot34$ and $67\cdot45$, respectively. The molecular conformation is
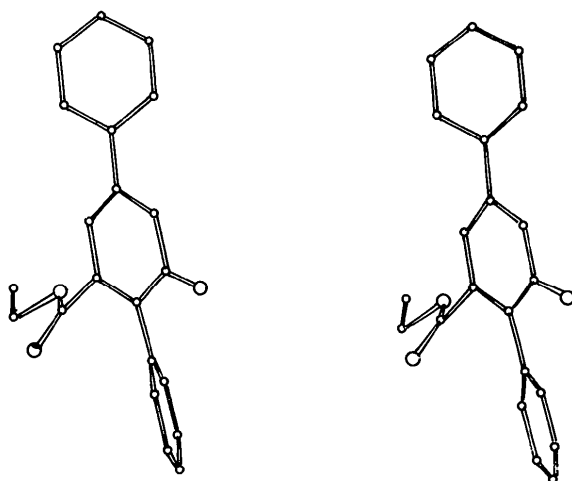

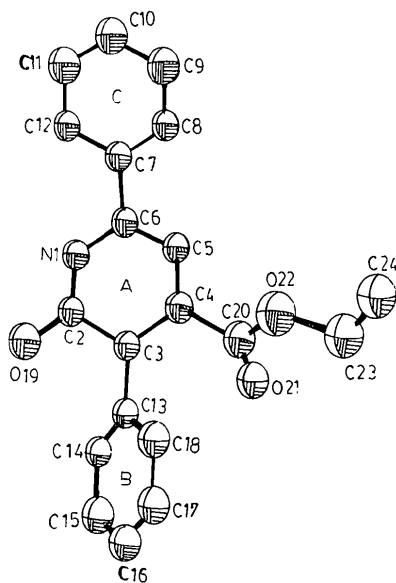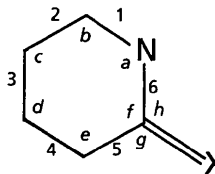Fig. 1. Stereoview of the molecule in the asymmetric unit.

Fig. 2. Drawing of the molecule showing thermal ellipsoids at the 50% probability level.

Table 3. *Comparison of protonated pyridine ring geometry*

Bond lengths (Å) and angles (°) are labelled according to the following layout:

1	2	3	4	5	6	Ref.	X
1·354 (3)	1·352 (4)	1·399 (4)	1·376 (4)	1·417 (4)	1·357 (3)	I	S
1·356 (3)	1·342 (3)	1·405 (3)	1·343 (3)	1·427 (3)	1·366 (3)	II	Cl
1·357 (4)	1·352 (6)	1·392 (6)	1·347 (6)	1·405 (6)	1·373 (4)	III	O
1·34 (3)	1·37 (3)	1·42 (3)	1·33 (3)	1·44 (3)	1·40 (3)	IV	O
1·359 (6)	1·381 (7)	1·40 (1)	1·356 (7)	1·440 (7)	1·371 (9)	V	O
1·365 (5)	1·367 (5)	1·405 (5)	1·368 (5)	1·444 (5)	1·378 (5)	VI	O

a	b	c	d	e	f	g	h	Ref.
124·6(2)	120·7(2)	118·0(3)	120·6(3)	120·9(2)	115·0(2)	124·3(2)	120·6(2)	I
124·4(3)	119·6(3)	119·6(3)	120·1(1)	121·3(3)	114·0(9)	125·2(3)	119·8(3)	II
124·0(3)	120·0(3)	118·0(3)	121·0(3)	121·0(3)	115·0(3)	126·0(3)	119·0(3)	III
125·1(2)	121·8(2)	116·0(2)	122·2(2)	122·3(2)	112·7(2)	126·0(2)	121·3(2)	IV
129·9(4)	118·0(6)	119·1(5)	122·9(5)	117·9(6)	117·2(4)	123·7(6)	119·2(5)	V
125·2(3)	117·9(3)	120·1(6)	121·9(3)	118·5(3)	116·4(3)	124·2(3)	119·4(3)	VI

Compounds and references: I 2-thiopyridone (Ohms, Guth, Kutoglu & Scheringer, 1982); II 5,5'-dichloro-3,3'-dinitro-2,2'-biphenyldiol (Hay & Mackay, 1981); III 6-chloro-2-hydroxypyridine–2-pyridone (Almlöf, Kvick & Olovsson, 1971); IV α-pyridone (Penfold, 1953); V 3-ethyl-6-phenyl-4-methoxycarbonyl-1H-2-pyridone (Nethaji, Vasantha Pattabhi & Gabe, 1989a); VI present work.

Table 4. *The carbonyl* C=O *and hydrogen-bond lengths* (Å) *in related compounds*

C=O	D—H···O	D···O	Ref.
1·262 (4)	O—H···O	2·570 (4)	I
1·250 (3)	N—H···O	2·796 (3)	II
1·242 (6)	N—H···O	2·82 (1)	III
1·244 (4)	N—H···O	2·808 (5)	IV

Compounds and references: I 6-chloro-2-hydroxypyridine–2-pyridone (Almlöf, Kvick & Olovsson, 1971); II 5-chloro-2-pyridone (Kvick & Booles, 1972); III 3,6-phenyl-4-methoxycarbonyl-1H-2-pyridone (Nethaji, Vasantha Pattabhi & Gabe, 1988a); IV present study.

stabilized by N—H···O and C—H···O types of hydrogen bonds. Centrosymmetrically related molecules form dimers about the centres of inversion and the hydrogen-bond formation is reminiscent of pyrimidine-purine base pairing in nucleotides. The hydrogen-bonding pattern is identical to the one observed in the related structure 3-ethyl-6-phenyl-4-methoxycarbonyl-1H-2-pyridone (Nethaji, Vasantha Pattabhi & Gabe, 1989a). The atom O(19) is hydrogen bonded to N(1) and C(12) forming a bifurcated hydrogen bond where one acceptor is hydrogen bonded to two donors and this type of hydrogen bond is commonly observed in urea complexes (Vaughan & Donohue, 1952). N(1)···O(19) = 2·808 (5) Å, N(1)—HN(1) = 1·1 (1) Å and N(1)—HN(1)···O(19) = 159 (6)°, and C(12)···O(19) =
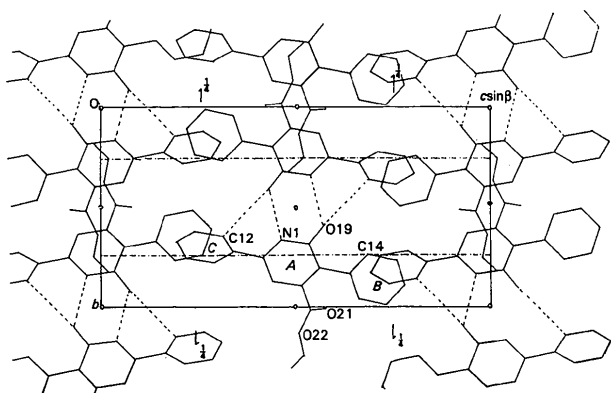
Fig. 3. Packing of the molecules in the unit cell projected down the *a* axis. Hydrogen bonds are shown as dotted lines.

3·192 (5) Å, C(12)—HC(12) = 0·91 (6) Å and C(12)—HC(12)···O(19) = 136 (4)° at the symmetry position −*x*, −*y*, −*z*. The hydrogen-bond geometry is shown in Fig. 3.

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Structure of 2-(2,6-Dimethoxyphenyl)-4*H*-1-benzopyran-4-one (2',6'-Dimethoxyflavone)

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Abstract. $C_{17}H_{14}O_4.C_2H_4O_2$, $M_r = 342.34$, triclinic, $P\bar{1}$, $a = 9.488$ (3), $b = 12.957$ (4), $c = 7.664$ (2) Å, $\alpha = 102.9$ (1), $\beta = 105.9$ (1), $\gamma = 91.1$ (1)°, $V = 880.0$ (1) Å³, $Z = 2$, $D_m = 1.35$, $D_x = 1.29$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.9$ cm⁻¹, $F(000) = 360$, $T = 293$ K, $R = 0.046$ for 1327 reflections. The dimethoxyphenyl ring at C2 is rotated 70.7° with respect to the fused two-ring system because of steric interaction between the methoxy groups at C2' and C6' and the H atom at C(3). The effect of the hydrogen bond with a molecule of solvent is investigated by comparison with a similar structure reported earlier.

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Introduction. Structures of bioflavonoids and their synthetic analogues have been investigated to try to explain their mechanism of action and their biological activity (Rossi, Rickles & Halpin, 1986; Cantrell, 1986). As part of our studies on hydroxy- and methoxyflavonoids (Wallet, Gaydou, Fadlane & Baldy, 1988) the structure of 2',6'-dimethoxyflavone (I) has been determined by single-crystal X-ray methods to provide conformational information.

Compound (I) was prepared by addition of excess thionyl chloride to 2,6-dimethoxybenzoic acid. The flask was mildly warmed until reaction ceased. After removal of unreacted thionyl chloride on a rotary evaporator, pyridine and *o*-hydroxyacetophenone were