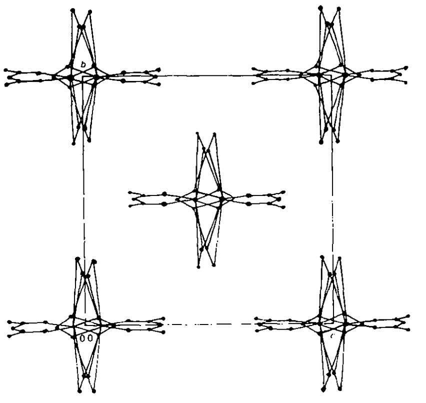
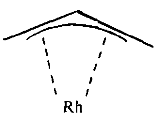
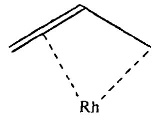


Fig. 2. A packing diagram viewed down **a**. F atoms are omitted.

Golding & Stofko, 1976). Structurally, the major effect is the opening of the cyclopropane ring, *i.e.* conversion of a bis(vinyl) to a bis(allyl) hydrocarbon; the C(13)–C(13′) distance is 2·25 Å. The resulting complex is similar to the bis(η-allyl)rhodium chloride dimer (McPartlin & Mason, 1967), but the presence of the bridgehead atom, C(14), produces a significant difference. In an ideal η-allyl, all three atoms are sp^2 hybridized, the p orbitals point towards the metal, and all five H atoms are to a first approximation in the plane of the three C atoms.* However, C(14), replacing one terminal H atom, is 0·80 Å out of the plane of C(11)–C(13). The p orbital on C(11)–C(12) can then only be weakly conjugated with that on C(13) [dihedral angle of 60° between C(11)–C(13) and C(13)–C(14)–C(13′)]; the bonding must largely go over from (*A*) to (*B*).



(*A*)



(*B*)

In keeping with this, the Rh–C(13) length, 2·09 Å, is considerably shorter than Rh–C(11) and Rh–C(12) at 2·26 and 2·23 Å. In contrast, if there is a two-carbon bridge, as in $C_{10}H_{16}Ru(PF_3)Cl_2$ (Hitchcock, Nixon & Sinclair, 1975), the atoms corresponding to C(14) are virtually in the allyl planes (by visual inspection of the published figure); the individual Rh–C lengths have not been published, but would be expected to be more uniform.

The packing (Fig. 2) is regular and dominated by F–F interactions.

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[*] In theory, this planarity could be affected by rehybridization from sp^2 to sp^3 if partly localized M–C σ-bonds are formed, while experimentally, in 2-methyl derivatives, the methyl group may be out of plane either towards or away from the metal atom (Marsh, Howard & Woodward, 1973).

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3-Pyridinesulphonic Acid

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Abstract. $C_5H_5NO_3S$, an antimetabolite of niacin, orthorhombic, *Pbca*; $a = 11{\cdot}449$ (2), $b = 14{\cdot}975$ (3), $c = 7{\cdot}186$ (1) Å, $V = 1232$ Å^3; $Z = 8$, $D_c = 1{\cdot}718$, $D_o = 1{\cdot}72$ g cm^{-3}; $M_r = 159{\cdot}2$. The structure was solved by heavy-atom and Fourier methods and refined by full-matrix least squares to $R = 11{\cdot}5\%$ for 910 in-

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[*] Contribution No. 439.

dependent reflexions. Layer-wise packing is observed and the structure is stabilized by hydrogen bonds of the NH · · · O type.

Introduction. Intensities were collected photographically with the multiple-film equi-inclination Weissenberg technique and Ni-filtered Cu *Kα* radiation ($\lambda = 1\cdot5418$ Å). About 900 reflexions (*hkl*, $l = 0$ to 6 and *hkl*, $h = 0$ to 2) were estimated visually by comparison with a standard set of spots prepared from the same crystal. They were corrected for Lorentz, polarization and spot-shape factors (Phillips, 1954) but not for absorption ($\mu t < 1$).

The position of the S atom was determined from a Patterson synthesis. The other atoms were located by a heavy-atom-phased Fourier synthesis in which only the reflexions with $|F_s| \geq \frac{1}{2}|F_o|$ were used. Structure factor calculations with all ten nonhydrogen atoms gave $R = 0\cdot37$. Block-diagonal least-squares refinement, first with isotropic then with anisotropic temperature factors, reduced R to 0·14. The five H atoms were then located from a difference map. An interesting feature was that the N atom appeared to be protonated. The H atoms were given the isotropic temperature factors of the atoms to which they were attached. Full-matrix least-squares refinement was carried out (Gantzel, Sparks & Trueblood, 1961) with anisotropic temperature factors for the nonhydrogen atoms and the weighting scheme of Cruickshank, Bujosa, Lovell & Truter (1961): $w = [a + |F_o| + b|F_o|^2]^{-1}$ with $a = 6$, $b = 0\cdot015$. The final R was 0·115.* The positions of the H atoms were not refined. Scattering factors were taken from *International Tables for X-ray Crystallography* (1962) and anomalous-dispersion corrections were applied for S. The final positional and thermal parameters of the nonhydrogen atoms are listed in Table 1. Table 2 gives the positions of the H atoms.

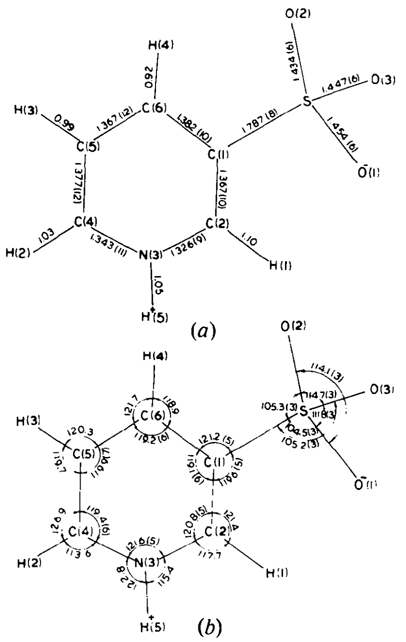

Fig. 1. (*a*) Bond lengths and (*b*) bond angles in 3-pyridinesulphonic acid. The e.s.d.'s are given in parentheses.

Discussion. The bond lengths and angles are illustrated in Fig. 1. They are similar to those found in nicotinic acid ($C_6H_5NO_2$) (Wright & King, 1953; Gupta & Kumar, 1975), of which 3-pyridinesulphonic acid is an antimetabolite. The S—C distance of 1·787 Å is close to both the average single-bond length quoted by Sutton (1958) and to the experimental values of 1·77 (Rae & Maslen, 1962), 1·782 (Jackobs & Sundaralingam, 1969) and 1·785 Å (Ammon, 1970).

The equation of the least-squares plane through the

Table 1. *Atomic positional* (×10⁴) *and anisotropic thermal* (×10⁴) *parameters for the nonhydrogen atoms, with e.s.d.'s in parentheses*

The temperature factor is of the form: $T = \exp[-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{13}hl + b_{23}kl)]$.

	x	y	z	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
S	3154 (2)	1203 (1)	−2247 (2)	53 (1)	32 (1)	92 (5)	−3 (2)	−4 (3)	4 (2)
O(1)	3665 (5)	544 (4)	−3488 (7)	78 (5)	44 (3)	114 (12)	14 (6)	−6 (12)	−14 (9)
O(2)	3192 (6)	2099 (4)	−2946 (9)	102 (6)	36 (3)	198 (16)	−4 (7)	−86 (16)	31 (10)
O(3)	2024 (5)	923 (5)	−1559 (9)	45 (5)	74 (4)	168 (14)	−25 (7)	−7 (12)	25 (12)
C(1)	4089 (6)	1202 (5)	−255 (10)	45 (5)	26 (3)	127 (16)	3 (6)	−14 (14)	−12 (10)
C(2)	3676 (7)	895 (6)	1410 (9)	61 (16)	39 (4)	69 (15)	3 (8)	14 (15)	8 (10)
N(3)	4329 (6)	935 (5)	2931 (9)	72 (6)	39 (3)	87 (13)	17 (7)	−5 (13)	9 (9)
C(4)	5405 (8)	1291 (6)	2904 (12)	71 (7)	42 (4)	149 (19)	17 (8)	−58 (17)	−16 (12)
C(5)	5858 (8)	1597 (6)	1248 (13)	56 (6)	44 (5)	199 (21)	5 (9)	−36 (18)	4 (14)
C(6)	5205 (7)	1550 (6)	−342 (11)	53 (5)	39 (4)	143 (17)	3 (8)	11 (16)	13 (12)

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31984 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

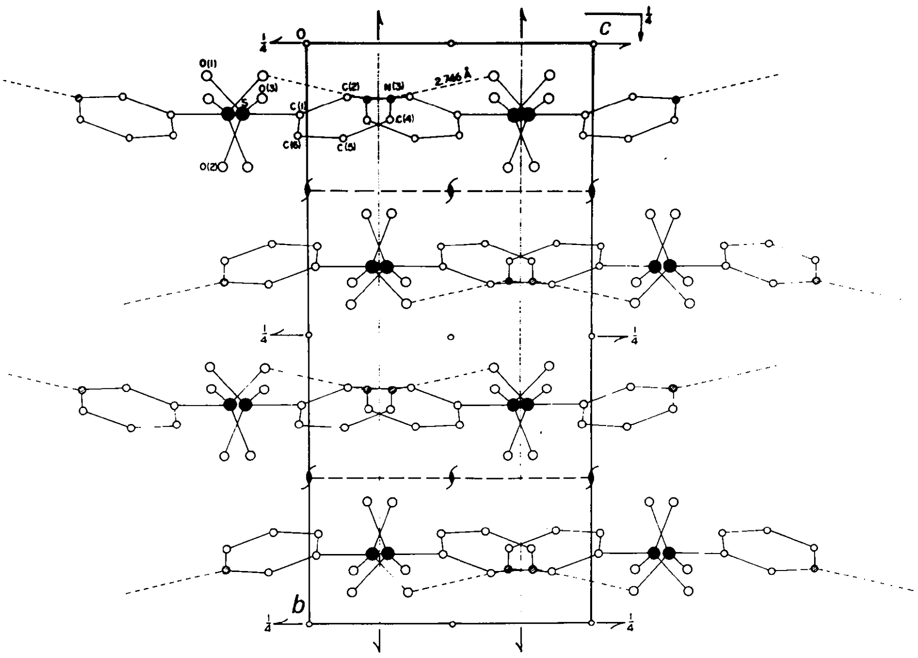

Fig. 2. The crystal structure projected along **a**. Hydrogen bonds are shown by broken lines.

Table 2. *Positional and thermal parameters of the hydrogen atoms*

	x	y	z	B (Å^2)
H(1)	0·2764	0·0639	0·1556	2·858
H(2)	0·5805	0·1306	0·4200	3·386
H(3)	0·6667	0·1833	0·1208	3·263
H(4)	0·5458	0·1792	−0·1433	2·887
H(5)	0·3917	0·0722	0·4167	2·677

ring atoms is $0{\cdot}3731X - 0{\cdot}9069Y - 0{\cdot}1953Z = 0{\cdot}1579$, where X, Y and Z refer to a, b and c.

The arrangement of molecules in the crystal is shown in Fig. 2. A layer-wise packing is observed. The structure is stabilized by hydrogen bonds of the NH···O type. There is only one active H atom in the molecule. This links O(1) to the (N)—H$^+$ of a translated molecule by a hydrogen bond of length 2·74 Å.

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