

liaisons se trouve dans le Tableau 2. La Fig. 1 montre une vue en perspective du composé avec la numérotation atomique. La Fig. 2 représente la structure cristalline.

**Littérature associée.** La structure du (méthyl-4 pipérazinyl-1)-10 pyrido[4,3-*b*][1,4]benzothiazépine a été réalisée dans le cadre de notre étude sur les récepteurs à dopamine. Voir par exemple la structure du (méthyl-1 pipérazinyl-4)-5 pyrido[2,3-*b*][1,5]benzothiazépine (Sbit, Dupont, Dideberg, Liégeois & Delarge, 1988), la structure de la clothiapine (Sbit, Dupont, Dideberg, Liégeois & Delarge, 1987) et la structure du maléate de méthyl-1 (pyrido[2,3-*f*][1,5]-benzoxazépiny-5)  $1H^+$ -pipérazinium (Dupont, Dideberg, Liégeois & Delarge, 1987). Des analogues hétérocycliques originaux sont en voie d'expérimentation.

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## Structure of 6-Oxo-1,6-dihydro-3,4'-bipyridine-5-carbonitrile Hydrogen Bromide

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**Abstract.** 5-Cyano-6-oxo-1,6-dihydro-3,4'-bipyridinium bromide,  $C_{11}H_8N_3O^+Br^-$ ,  $M_r = 278.11$ , triclinic,  $P\bar{1}$ ,  $a = 7.827$  (2),  $b = 9.796$  (2),  $c = 15.548$  (4) Å,  $\alpha = 76.53$  (2),  $\beta = 75.40$  (2),  $\gamma = 74.36$  (2)°,  $V = 1093.4$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.689$  Mg m<sup>-3</sup>,  $\lambda(Mo K\alpha) = 0.71073$  Å,  $\mu = 3.70$  mm<sup>-1</sup>,  $F(000) = 552$ ,  $T = 293$  K,  $R = 0.084$  for 3989 observed reflections. The bipyridinium ions are planar, the C(2)—C(1)—C(1')—C(2') torsion angles being  $-3.3$  (10) and  $2.2$  (11)° for molecules (1) and (2), respectively. The symmetry-independent bipyridinium ions are assembled in separate chains by hydrogen-bonding Br bridges [respective distances: Br(1)⋯N(3) 3.227 (6); Br(1)⋯N(4') 3.374 (6); Br(2)⋯N(3\*) 3.263 (5); Br(2)⋯N(4'\*) 3.540 (7) Å] and by forming N⋯O hydrogen bonds between pyridinium NH groups and keto O atoms, the N(4')⋯O(4) and N(4'\*)⋯O(4') distances being 2.835 (7) and 2.706 (7) Å, respectively. These chains are connected by the series of stacking interactions with an average C⋯C distance of 3.5 Å.

**Experimental.** Samples were obtained from Sterling-Winthrop. Crystals from the H<sub>2</sub>O/EtOH/HBr solution; cell parameters determined from 25 reflections  $21.99 < 2\theta < 29.58^\circ$ ; Syntex P3 diffractometer, Nb filter, Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å;  $\omega$ - $2\theta$  scan; 5044 independent reflections  $4 < 2\theta < 55^\circ$  ( $0 < h < 10$ ,  $-11 < k < 11$ ,  $-19 < l < 19$ ) measured for a  $0.38 \times 0.35 \times 0.10$  mm rectangular crystal; six standards measured every 138 reflections revealed no significant intensity variation, no absorption or extinction corrections; 3989 observed reflections [ $I > 3\sigma(I)$ ]; direct methods used *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and *NQUEST* (De Titta, Edmonds, Langs & Hauptman, 1975),  $\sum w(|F_o| - |F_c|)^2$  minimized with full-matrix least squares,  $w = 1/\sigma^2(F)$ . H-atom positions from geometry, two H atoms from pyridinium ions missing in the structure, only non-H atom parameters refined (anisotropically). Final  $|\Delta/\sigma| < 0.01$ ,  $\Delta\rho_{\max} = 2.61$  and  $\Delta\rho_{\min} = -1.65$  e Å<sup>-3</sup>,  $R = 0.084$ ,  $wR = 0.078$  and  $S = 3.676$ . The large  $R$  value is a result of the crystal thickness and the absorption effects of Br<sup>-</sup>. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final

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Table 1. Fractional atomic coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\times 10^2 \text{ \AA}^2$ )
$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

|        | <i>x</i>   | <i>y</i>  | <i>z</i> | <i>B</i> <sub>eq</sub> |
|--------|------------|-----------|----------|------------------------|
| Br(1)  | 6806 (1)   | 1068 (1)  | 470 (1)  | 419 (3)                |
| Br(2)  | 8166 (1)   | 3761 (1)  | 4654 (1) | 323 (2)                |
| C(1)   | 2491 (9)   | 4808 (6)  | 2613 (4) | 232 (19)               |
| C(2)   | 3504 (10)  | 4139 (7)  | 1915 (4) | 290 (21)               |
| N(3)   | 4766 (9)   | 2903 (6)  | 2058 (4) | 316 (18)               |
| C(4)   | 5178 (10)  | 2200 (7)  | 2884 (4) | 275 (21)               |
| C(5)   | 4060 (10)  | 2871 (7)  | 3619 (4) | 247 (20)               |
| C(6)   | 2792 (10)  | 4118 (6)  | 3496 (4) | 249 (20)               |
| O(4)   | 6383 (8)   | 1098 (5)  | 2935 (3) | 403 (17)               |
| C(51)  | 4280 (11)  | 2176 (7)  | 4516 (5) | 311 (22)               |
| N(52)  | 4476 (11)  | 1642 (7)  | 5231 (4) | 470 (25)               |
| C(1')  | 1143 (9)   | 6177 (6)  | 2454 (4) | 230 (19)               |
| C(2')  | 953 (11)   | 6850 (7)  | 1574 (5) | 322 (22)               |
| C(3')  | -301 (11)  | 8145 (7)  | 1438 (5) | 374 (25)               |
| N(4')  | -1310 (8)  | 8757 (6)  | 2133 (4) | 309 (19)               |
| C(5')  | -1125 (10) | 8151 (7)  | 2978 (5) | 322 (23)               |
| C(6')  | 65 (10)    | 6851 (7)  | 3164 (5) | 277 (21)               |
| C(1*)  | 4353 (9)   | 8040 (6)  | 2449 (4) | 225 (18)               |
| C(2*)  | 4940 (10)  | 7072 (7)  | 3171 (4) | 249 (20)               |
| N(3*)  | 6192 (8)   | 5872 (5)  | 3056 (4) | 251 (17)               |
| C(4*)  | 6992 (10)  | 5447 (7)  | 2234 (4) | 252 (20)               |
| C(5*)  | 6382 (10)  | 6458 (7)  | 1475 (4) | 274 (20)               |
| C(6*)  | 5118 (10)  | 7690 (7)  | 1581 (4) | 259 (20)               |
| O(4*)  | 8124 (8)   | 4304 (5)  | 2186 (3) | 385 (17)               |
| C(51*) | 7163 (13)  | 6075 (8)  | 606 (5)  | 440 (28)               |
| N(52*) | 7776 (14)  | 5760 (9)  | -92 (5)  | 715 (35)               |
| C(1'*) | 2945 (10)  | 9361 (6)  | 2591 (4) | 231 (19)               |
| C(2'*) | 2195 (12)  | 9711 (7)  | 3451 (5) | 380 (25)               |
| C(3'*) | 878 (12)   | 10941 (8) | 3558 (5) | 424 (27)               |
| N(4'*) | 286 (9)    | 11833 (6) | 2859 (5) | 359 (21)               |
| C(5'*) | 944 (12)   | 11543 (7) | 2022 (5) | 389 (26)               |
| C(6'*) | 2312 (11)  | 10334 (7) | 1868 (5) | 352 (23)               |

Table 2. Bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) with their *e.s.d.*'s for the title compound

|                   | Molecule (1) | Molecule (2) |
|-------------------|--------------|--------------|
| C(1)—C(2)         | 1.361 (9)    | 1.378 (9)    |
| C(1)—C(6)         | 1.429 (9)    | 1.411 (9)    |
| C(1)—C(1')        | 1.479 (8)    | 1.477 (8)    |
| C(2)—N(3)         | 1.353 (8)    | 1.328 (8)    |
| C(4)—N(3)         | 1.380 (9)    | 1.379 (8)    |
| C(4)—C(5)         | 1.427 (9)    | 1.444 (9)    |
| C(4)—O(4)         | 1.229 (8)    | 1.231 (7)    |
| C(5)—C(6)         | 1.359 (8)    | 1.352 (9)    |
| C(5)—C(51)        | 1.433 (10)   | 1.428 (10)   |
| C(51)—N(52)       | 1.143 (10)   | 1.148 (11)   |
| C(1')—C(2')       | 1.399 (10)   | 1.399 (10)   |
| C(1')—C(6')       | 1.392 (9)    | 1.395 (9)    |
| C(2')—C(3')       | 1.388 (9)    | 1.370 (10)   |
| C(3')—N(4')       | 1.329 (10)   | 1.324 (10)   |
| C(5')—N(4')       | 1.336 (10)   | 1.340 (11)   |
| C(5')—C(6')       | 1.378 (9)    | 1.388 (9)    |
| C(2)—C(1)—C(6)    | 116.7 (5)    | 117.1 (5)    |
| C(2)—C(1)—C(1')   | 121.0 (5)    | 120.7 (5)    |
| C(6)—C(1)—C(1')   | 122.3 (5)    | 122.1 (5)    |
| C(1)—C(2)—N(3)    | 121.0 (5)    | 121.6 (5)    |
| C(2)—N(3)—C(4)    | 125.6 (5)    | 124.8 (5)    |
| N(3)—C(4)—C(5)    | 113.2 (5)    | 113.8 (5)    |
| N(3)—C(4)—O(4)    | 120.3 (5)    | 120.8 (5)    |
| C(5)—C(4)—O(4)    | 126.5 (5)    | 125.3 (5)    |
| C(4)—C(5)—C(6)    | 122.4 (5)    | 122.0 (5)    |
| C(4)—C(5)—C(51)   | 118.0 (5)    | 116.3 (6)    |
| C(6)—C(5)—C(51)   | 119.5 (5)    | 121.7 (6)    |
| C(1)—C(6)—C(5)    | 120.9 (5)    | 120.7 (5)    |
| C(5)—C(6)—N(3)    | 178.5 (6)    | 179.3 (7)    |
| C(1)—C(1')—C(2')  | 120.4 (5)    | 122.1 (5)    |
| C(1)—C(1')—C(6')  | 121.5 (5)    | 121.3 (5)    |
| C(2')—C(1')—C(6') | 118.1 (5)    | 116.7 (5)    |
| C(1')—C(2')—C(3') | 119.5 (6)    | 120.5 (6)    |
| C(2')—C(3')—N(4') | 120.7 (6)    | 121.5 (6)    |
| C(3')—N(4')—C(5') | 120.9 (5)    | 120.5 (6)    |
| N(4')—C(5')—C(6') | 121.5 (6)    | 120.6 (6)    |
| C(1')—C(6')—C(5') | 119.3 (5)    | 120.2 (6)    |

Table 3. Geometry of the hydrogen bonds

| D—H...A              | D—H ( $\text{\AA}$ ) | H...A ( $\text{\AA}$ ) | D...A ( $\text{\AA}$ ) | D—H...A ( $^\circ$ ) |
|----------------------|----------------------|------------------------|------------------------|----------------------|
| N(3)—H(3)...Br(1')   | 1.07                 | 2.20                   | 3.227 (6)              | 162                  |
| N(4')...Br(1')       |                      |                        | 3.374 (6)              |                      |
| N(4')...O(4')        |                      |                        | 2.835 (7)              |                      |
| N(3*)—H(3*)...Br(2') | 0.90                 | 2.38                   | 3.263 (5)              | 172                  |
| N(4'*)...Br(2')      |                      |                        | 3.540 (7)              |                      |
| N(4'*)...O(4'*)      |                      |                        | 2.706 (7)              |                      |

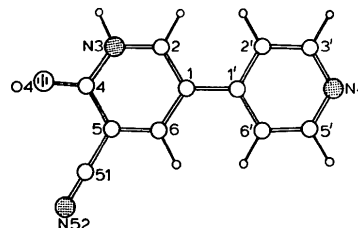
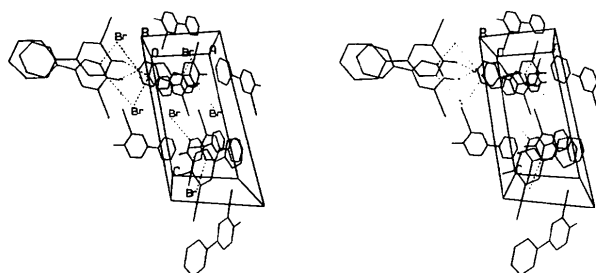
Symmetry codes: (i) *x*, *y*, *z*; (ii)  $-1 + x$ ,  $1 + y$ , *z*.

Fig. 1. The 6-oxo-1,6-dihydro-3,4'-bipyridine-5-carbonitrile molecule with the numbering scheme.

Fig. 2. Stereoview of the packing diagram projected down the *b* axis. Hydrogen bonds are indicated by broken lines.

non-H-atom fractional coordinates and equivalent isotropic thermal parameters are listed in Table 1, bond lengths and angles are listed in Table 2 and hydrogen-bond geometry is summarized in Table 3.\* Other programs used: those from Enraf-Nonius package, VAX8600 computer. The atomic numbering scheme is shown in Fig. 1 and the packing diagram in Fig. 2.

**Related literature.** The crystal structures of other 3,4'-bipyridin-6(1*H*)-one derivatives and their biological function have been determined by Cody (1987); Cody, Suwińska & Wojtczak (1991) and Cody & Wojtczak (1991), and references cited therein.

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and details of the best planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53900 (38 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of 2',4'-Dihydroxy-4,6'-dimethoxy- $\alpha,\beta$ -dihydrochalcone

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**Abstract.** C<sub>17</sub>H<sub>18</sub>O<sub>5</sub>,  $M_r = 302$ , monoclinic,  $P2_1/c$ ,  $a = 4.856$  (3),  $b = 28.896$  (7),  $c = 10.776$  (3) Å,  $\beta = 98.04$  (4)°,  $V = 1497$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.34$  g cm<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 0.922$  cm<sup>-1</sup>,  $F(000) = 640$ , room temperature,  $R = 0.068$ ,  $wR = 0.074$  for 792 observed reflections [ $I \geq 2.5\sigma(I)$ ]. The phenyl rings are planar within experimental accuracy; the angle between the normals to the best least-squares planes of the phenyl rings is 81.49 (9)°. Intermolecular and intramolecular hydrogen bonds are present: O1...H3'—O3' ( $x - 1, \frac{1}{2} - y, \frac{1}{2} + z$ ), 2.77 (1) Å, O2—H2...O1, 2.50 (1) Å; this interaction may contribute to the low value of the O1—C1—C2—C3 [ $-5.42$  (9)°] and O1—C1—C11—C12 [ $5.63$  (9)°] torsion angles. The molecular structure found is in total agreement with spectroscopic results.

**Experimental.** Transparent colourless flat prism of dimensions 0.20 × 0.08 × 0.15 mm, Enraf-Nonius CAD-4 diffractometer, Mo  $K\alpha$  radiation, graphite monochromator. Unit-cell parameters were determined by least squares from 25 reflections with  $3 \leq \theta \leq 13^\circ$ ; 5191 reflections collected, 2632 unique, 792 considered observed with  $I \geq 2.5\sigma(I)$ ;  $\omega$ - $2\theta$  scan mode,  $hkl$  range:  $-5 \leq h \leq 5$ ,  $-34 \leq k \leq 34$ ,  $0 \leq l \leq 12$ ,  $\theta \leq 25^\circ$ . Three standard reflections monitored every 50 measurements showed no significant decay in intensity. Lorentz-polarization corrections, but no absorption correction. Structure solved by direct

methods (*MULTAN11/84*; Main, Germain & Woolfson, 1984). Anisotropic full-matrix least-squares refinement (on  $F$ ) for non-H atoms (*SHELX76*; Sheldrick, 1976). Hydroxy H atoms were found by difference Fourier synthesis and included in the refinement, and the remaining H atoms were included at calculated positions in the final cycle and refined; two different isotropic temperature factors, one for methyl and one for non-methyl H atoms, were refined to final values  $U = 0.106$  (16) and 0.121 (13) Å<sup>2</sup>, respectively;  $R = 0.068$ ,  $wR = 0.074$ ,  $w = 1.2689/[\sigma^2(F) + 0.002575F^2]$  for 792 observed reflections and 207 variables. Final  $(\Delta/\sigma)_{\max} = 0.11$ ,  $S = 1.17$ . Largest peaks in final  $\Delta F$  map:  $-0.24$  and  $0.19$  e Å<sup>-3</sup>. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Plots were made with *PLUTO* (Motherwell & Clegg, 1978). All calculations were performed on a MicroVAX II computer.

The atomic parameters are given in Table 1.† A view of the molecule with the atomic numbering scheme is shown in Fig. 1; the molecular packing is shown in Fig. 2. Table 2 gives bond distances and angles.

† Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, torsion angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53935 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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