

plan moyen est intermédiaire entre celle observée dans les dicéoptène- α [$Q = 0,043$ (3)Å] et $-\epsilon$ [$Q = 0,122$ (3)Å]. L'angle entre les normales aux plans moyens $P1-P2[C(1)C(7)C(8)O(3)] = 1,4$ (1) $^\circ$ (8 à 14 $^\circ$ dans les dicéoptène- α et $-\epsilon$). La cohésion du cristal est assurée uniquement par des liaisons de van der Waals.

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Structures of 1-Phenyl-2,3,4-tris(trifluoromethyl)pyrrolo[3,2-*c*]quinoline (1) and 2-Fluoro-3-pentafluoroethyl-1-phenyl-2,3,4-tris(trifluoromethyl)-2,3-dihydropyrrolo[3,2-*c*]quinoline (2)

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Abstract. (1) C₂₀H₉F₉N₂, $M_r = 448.29$, monoclinic, $P2_1/c$, $a = 10.044$ (3), $b = 19.452$ (5), $c = 10.209$ (3) Å, $\beta = 117.78$ (2) $^\circ$, $V = 1764.7$ (10) Å³, $Z = 4$, $D_x = 1.687$ Mg m⁻³, $\lambda(\text{Mo } K\alpha_1) = 0.70926$ Å, $\mu = 0.18$ mm⁻¹, $F(000) = 896$, room temperature, $R = 0.072$ for 1940 independent observed reflections. (2) C₂₂H₉F₁₅N₂, $M_r = 586.30$, orthorhombic, $P2_12_12_1$, $a = 11.381$ (5), $b = 19.110$ (6), $c = 9.982$ (5) Å, $V = 2170.9$ (15) Å³, $Z = 4$, $D_x = 1.794$ Mg m⁻³, $\lambda(\text{Mo } K\alpha_1) = 0.70926$ Å, $\mu = 0.22$ mm⁻¹, $F(000) = 1160$, room temperature, $R = 0.054$ for 1919 independent observed reflections. The lone electron pair of N(1) for (1) takes part in conjugation (the pyrrole and quinoline rings form a π -conjugated system), but there is no conjugated system in the pyrrole ring of (2). The bond lengths of C–C and C–F on C(2) and C(3) in (2) are longer than the normal values, probably owing to steric hindrance.

Introduction. Compound (1) is a minor product of the ring-closing reaction of 2-phenylimino-3-phenylimino-fluoromethylene-4-trifluoromethylperfluorohexene-[3] with aniline in dimethylformamide at 393–433 K in the presence of KF, and compound (2) is obtained by the reaction of 5-phenylimino-4-phenyliminomethylene-3-

trifluoromethyl-3-pentafluoroethylperfluorohexane with dimethylformamide at 393–413 K (Chen, Wu & Wang, 1984). Such a ring-closing reaction, in which a nitrogen atom attacks an intramolecular inert difluoromethylene group, has rarely been reported. The determination of both structures of these heterocycles will provide a sound basis for exploring the reaction mechanism.

Experimental. Colourless crystals from *n*-hexane, D_m not determined; RASA-IIS diffractometer, $\omega/2\theta$ scan, $3 < 2\theta < 52^\circ$ for (1) and $3 < 2\theta < 56^\circ$ for (2), graphite-monochromated Mo $K\alpha_1$ radiation; lattice parameters from least-squares refinement of 12 medium-angle reflections [$12.57 \leq 2\theta \leq 33.08^\circ$ for (1) and $21.98 \leq 2\theta \leq 33.05^\circ$ for (2)]; no significant variation in intensity for three standard reflections; Lorentz and polarization corrections, no absorption correction applied; *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), manual choice of starting reflections on the bottom of the convergence map.

(1) Systematic absences $k + l = 2n + 1$ for $h0l$ and $k = 2n + 1$ for $0k0$; space group $P2_1/c$; 3496 independent reflections ($0 \leq h \leq 12$, $0 \leq k \leq 24$,

$-12 \leq l \leq 12$), 1940 with $F_o > 2\sigma(F_o)$; the F atoms are disordered as revealed from difference-Fourier analysis. The occupancy factor for every F atom was assigned so that the F atoms in pairs have nearly the same thermal parameters; H atoms were located in a difference Fourier map and refined; block-diagonal least-squares refinement on F (Ashida, 1967); $\sum w(|F_o| - |F_c|)^2$ minimized, $w^{-1} = \sigma^2(F)$, $R = 0.072$, $wR = 0.065$, $S = 2.1$, 389 parameters refined, $\Delta/\sigma < 0.25$, maximum absolute height $0.3 \text{ e } \text{Å}^{-3}$ in final difference Fourier map.

(2) Systematic absences $h = 2n + 1$ for $h00$, $k = 2n + 1$ for $0k0$ and $l = 2n + 1$ for $00l$; space group $P2_12_12_1$; 2987 independent reflections ($0 \leq h \leq 15$, $0 \leq k \leq 25$, $0 \leq l \leq 13$), 1919 with $F_o > 2\sigma(F_o)$; the CF_3 on the quinoline ring is disordered. The occupancy factor was assigned so that the F atoms in pairs have nearly the same thermal parameters; H atoms were located in a difference Fourier map and refined; full-matrix least-squares refinement on F using *SHELX76* (Sheldrick, 1976); $\sum w(|F_o| - |F_c|)^2$ minimized, $w^{-1} = 1.667/[\sigma^2(F) + 0.0005F^2]$, $R = 0.054$, $wR = 0.057$, $S = 2.6$, 416 parameters refined, $\Delta/\sigma < 0.11$, maximum absolute height $0.2 \text{ e } \text{Å}^{-3}$ in final difference Fourier map. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Sizes of crystals used: (1) $0.4 \times 0.4 \times 0.6 \text{ mm}$; (2) $0.3 \times 0.4 \times 0.7 \text{ mm}$.

Tables 1 and 2 list atomic positions for compounds (1) and (2) respectively, Table 3 lists interatomic distances and bond angles, and Table 4 lists torsion angles.*

Discussion. Figs. 1 and 2 are *ORTEP* drawings (Johnson, 1976) of compounds (1) and (2) respectively.

The pyrrole ring in (1) is coplanar with the quinoline moiety, and the lone electron pair of N(1) takes part in conjugation, forming a π -conjugated system with the quinoline ring. There is no conjugated system in the pyrrole ring of (2). The quinoline rings of (1) and (2) exhibit no significant difference. N(2) is an sp^2 hybrid atom. Its p_z electron forms with the p_z electron of C a π -bond conjugation, and because of the electron-withdrawing group CF_3 , the bond length N(2)—C(2') is shorter than N(2)—C(6). The dihedral angles between the planes of the phenyl and quinoline rings are 92° and 85° for (1) and (2) respectively, so they are approximately perpendicular to each other. The bond lengths of C—C and C—F on C(2) and C(3) in (2) are longer than

* Lists of structure factors, anisotropic thermal parameters, atomic parameters for H atoms, bond lengths involving H atoms, bond angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44195 (35 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 and B_{eq} values for compound (1)

$$B_{\text{eq}} = 4[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab(\cos\gamma)B_{12} + ac(\cos\beta)B_{13} + bc(\cos\alpha)B_{23}]/3.$$

| | <i>x</i> | <i>y</i> | <i>z</i> | $B_{\text{eq}}(\text{Å}^2)$ |
|-------|------------|-------------|------------|-----------------------------|
| N(1) | 0.6752 (4) | 0.1006 (2) | 0.8813 (4) | 3.3 (2) |
| C(2) | 0.8101 (5) | 0.1235 (2) | 0.9930 (5) | 3.5 (2) |
| C(3) | 0.8787 (5) | 0.0726 (3) | 1.0913 (5) | 3.8 (2) |
| C(4) | 0.7834 (5) | 0.0127 (2) | 1.0390 (5) | 3.2 (2) |
| C(5) | 0.6570 (5) | 0.0329 (2) | 0.9084 (5) | 3.0 (2) |
| C(2') | 0.7853 (5) | -0.0577 (2) | 1.0820 (5) | 3.5 (2) |
| N(2) | 0.6742 (4) | -0.1000 (2) | 1.0112 (4) | 3.6 (2) |
| C(6) | 0.5489 (5) | -0.0783 (2) | 0.8869 (5) | 3.4 (2) |
| C(7) | 0.4337 (6) | -0.1275 (2) | 0.8201 (6) | 4.0 (2) |
| C(8) | 0.3053 (6) | -0.1106 (3) | 0.6940 (6) | 4.2 (2) |
| C(9) | 0.2900 (6) | -0.0455 (3) | 0.6348 (6) | 4.3 (2) |
| C(10) | 0.3997 (5) | 0.0028 (2) | 0.6974 (5) | 3.9 (2) |
| C(11) | 0.5341 (5) | -0.0118 (2) | 0.8276 (5) | 3.0 (2) |
| C(12) | 0.5683 (5) | 0.1395 (2) | 0.7536 (5) | 3.3 (2) |
| C(13) | 0.5779 (6) | 0.1348 (3) | 0.6242 (6) | 4.5 (3) |
| C(14) | 0.4696 (7) | 0.1684 (3) | 0.5010 (7) | 5.2 (3) |
| C(15) | 0.3574 (6) | 0.2053 (3) | 0.5086 (6) | 4.6 (2) |
| C(16) | 0.3505 (6) | 0.2103 (3) | 0.6406 (6) | 4.5 (3) |
| C(17) | 0.4579 (6) | 0.1767 (2) | 0.7662 (6) | 3.9 (2) |
| C(18) | 0.8576 (6) | 0.1974 (3) | 0.9918 (7) | 5.3 (3) |
| C(19) | 1.0204 (7) | 0.0821 (3) | 1.2317 (7) | 6.3 (3) |
| C(20) | 0.9171 (6) | -0.0925 (3) | 1.2114 (6) | 5.1 (3) |
| F(1) | 0.8128 (5) | 0.2209 (2) | 0.8544 (4) | 5.7 (2) |
| F(2) | 1.0069 (5) | 0.2015 (2) | 1.0528 (5) | 8.3 (3) |
| F(3) | 0.7965 (6) | 0.2390 (2) | 1.0469 (6) | 10.3 (3) |
| F(4) | 1.1425 (4) | 0.0727 (3) | 1.2277 (5) | 7.6 (3) |
| F(5) | 1.0167 (4) | 0.0422 (2) | 1.3415 (4) | 6.0 (2) |
| F(6) | 1.0216 (5) | 0.1468 (2) | 1.2885 (4) | 6.6 (2) |
| F(7) | 0.9217 (5) | -0.0831 (2) | 1.3383 (4) | 5.7 (2) |
| F(8) | 1.0496 (4) | -0.0692 (2) | 1.2182 (5) | 5.9 (2) |
| F(9) | 0.9167 (5) | -0.1595 (2) | 1.1891 (5) | 6.7 (2) |

Table 2. Fractional atomic coordinates and B_{eq} values for compound (2)

$$B_{\text{eq}} = 8\pi^2(U_{11} + U_{22} + U_{33} + 2U_{12} + 2U_{13} + 2U_{23})/3.$$

| | <i>x</i> | <i>y</i> | <i>z</i> | $B_{\text{eq}}(\text{Å}^2)$ |
|-------|-------------|-------------|-------------|-----------------------------|
| N(1) | 0.4519 (4) | 0.1675 (2) | 0.9216 (4) | 3.6 (2) |
| C(2) | 0.3864 (5) | 0.1821 (3) | 1.0387 (5) | 3.3 (3) |
| C(3) | 0.4261 (4) | 0.1251 (3) | 1.1426 (6) | 3.4 (3) |
| C(4) | 0.4751 (4) | 0.0680 (2) | 1.0496 (5) | 2.7 (2) |
| C(5) | 0.4938 (4) | 0.0987 (2) | 0.9256 (5) | 2.8 (2) |
| C(2') | 0.5132 (4) | -0.0015 (3) | 1.0653 (5) | 3.9 (3) |
| N(2) | 0.5652 (4) | -0.0368 (2) | 0.9693 (5) | 4.2 (2) |
| C(6) | 0.5859 (4) | -0.0055 (3) | 0.8498 (5) | 3.0 (3) |
| C(7) | 0.6456 (5) | -0.0476 (3) | 0.7522 (6) | 4.0 (3) |
| C(8) | 0.6711 (5) | -0.0210 (4) | 0.6302 (6) | 5.5 (4) |
| C(9) | 0.6430 (6) | 0.0510 (4) | 0.6044 (6) | 7.7 (4) |
| C(10) | 0.5855 (6) | 0.0913 (3) | 0.6940 (6) | 7.7 (3) |
| C(11) | 0.5538 (5) | 0.0640 (3) | 0.8217 (5) | 3.7 (3) |
| C(12) | 0.4219 (5) | 0.2027 (3) | 0.7970 (6) | 3.9 (3) |
| C(13) | 0.3247 (6) | 0.1827 (3) | 0.7271 (6) | 4.3 (3) |
| C(14) | 0.2977 (7) | 0.2172 (4) | 0.6076 (7) | 5.3 (4) |
| C(15) | 0.3656 (9) | 0.2692 (4) | 0.5619 (6) | 9.4 (5) |
| C(16) | 0.4653 (8) | 0.2883 (3) | 0.6326 (8) | 8.5 (4) |
| C(17) | 0.4950 (7) | 0.2555 (3) | 0.7523 (7) | 6.0 (3) |
| C(18) | 0.3932 (7) | 0.2627 (3) | 1.0724 (7) | 5.3 (4) |
| C(19) | 0.5318 (6) | 0.1488 (4) | 1.2291 (6) | 3.7 (4) |
| C(20) | 0.4994 (7) | -0.0488 (4) | 1.1866 (7) | 8.2 (4) |
| C(21) | 0.3197 (5) | 0.1075 (3) | 1.2395 (6) | 5.6 (3) |
| C(22) | 0.2199 (6) | 0.0593 (3) | 1.1877 (8) | 5.8 (4) |
| F(1) | 0.4997 (4) | 0.2859 (2) | 1.0712 (5) | 5.2 (3) |
| F(2) | 0.3311 (4) | 0.2975 (2) | 0.9830 (4) | 8.6 (3) |
| F(3) | 0.3440 (4) | 0.2773 (2) | 1.1901 (4) | 7.6 (3) |
| F(4) | 0.6152 (3) | 0.1755 (2) | 1.1501 (4) | 3.8 (2) |
| F(5) | 0.5034 (3) | 0.1990 (2) | 1.3171 (4) | 3.7 (2) |
| F(6) | 0.5790 (4) | 0.0966 (2) | 1.2954 (4) | 6.9 (2) |
| F(7) | 0.4614 (13) | -0.1112 (4) | 1.1533 (9) | 8.9 (6) |
| F(8) | 0.4252 (10) | -0.0282 (5) | 1.2780 (9) | 15.8 (6) |
| F(9) | 0.6016 (9) | -0.0616 (8) | 1.2469 (14) | 13.1 (8) |
| F(10) | 0.2676 (3) | 0.1720 (2) | 1.0158 (3) | 4.5 (2) |
| F(11) | 0.3625 (4) | 0.0782 (2) | 1.3524 (4) | 8.3 (2) |
| F(12) | 0.2668 (3) | 0.1664 (2) | 1.2795 (4) | 6.5 (2) |
| F(13) | 0.2470 (4) | 0.0224 (2) | 1.0809 (5) | 4.6 (3) |
| F(14) | 0.1882 (4) | 0.0159 (3) | 1.2847 (6) | 10.4 (3) |
| F(15) | 0.1254 (3) | 0.0947 (3) | 1.1591 (6) | 8.5 (3) |

the normal values, and some bond angles deviate significantly from normal. The deviation results primarily from steric hindrance between groups.

All the trifluoromethyl groups in (1) and only one in (2) are disordered; they are probably situated in some

conformers due to low torsion energy. The bond lengths of disordered groups are stereochemically unrealistic. The structures consist of discrete molecules held together by van der Waals forces.

Table 3. *Interatomic distances (Å) and valence angles (°)*

| | Compound (1) | Compound (2) |
|----------------|--------------|--------------|
| N(1)—C(2) | 1.376 (7) | 1.414 (6) |
| C(2)—C(3) | 1.349 (8) | 1.571 (7) |
| C(3)—C(4) | 1.442 (8) | 1.537 (7) |
| C(4)—C(5) | 1.402 (8) | 1.386 (7) |
| C(5)—N(1) | 1.375 (7) | 1.399 (6) |
| C(4)—C(2') | 1.436 (8) | 1.405 (7) |
| C(2')—N(2) | 1.300 (8) | 1.313 (6) |
| N(2)—C(6) | 1.373 (8) | 1.355 (6) |
| C(6)—C(11) | 1.407 (8) | 1.405 (7) |
| C(6)—C(7) | 1.409 (9) | 1.435 (7) |
| C(7)—C(8) | 1.372 (9) | 1.351 (8) |
| C(8)—C(9) | 1.381 (9) | 1.436 (9) |
| C(9)—C(10) | 1.360 (9) | 1.350 (8) |
| C(10)—C(11) | 1.413 (8) | 1.424 (8) |
| C(11)—C(5) | 1.418 (8) | 1.408 (7) |
| N(1)—C(12) | 1.457 (7) | 1.455 (6) |
| C(12)—C(13) | 1.371 (9) | 1.362 (8) |
| C(13)—C(14) | 1.385 (10) | 1.396 (9) |
| C(14)—C(15) | 1.369 (10) | 1.339 (11) |
| C(15)—C(16) | 1.385 (9) | 1.385 (11) |
| C(16)—C(17) | 1.395 (9) | 1.391 (10) |
| C(17)—C(18) | 1.380 (9) | 1.382 (8) |
| C(2)—C(18) | 1.516 (10) | 1.579 (8) |
| C(3)—C(19) | 1.488 (10) | 1.549 (8) |
| C(2')—C(20) | 1.525 (9) | 1.520 (8) |
| C(3)—C(21) | | 1.585 (8) |
| C(21)—C(22) | | 1.552 (9) |
| C(18)—F(1) | 1.337 (9) | 1.290 (8) |
| C(18)—F(2) | 1.331 (10) | 1.318 (8) |
| C(18)—F(3) | 1.292 (10) | 1.331 (7) |
| C(19)—F(4) | 1.260 (10) | 1.336 (7) |
| C(19)—F(5) | 1.377 (9) | 1.341 (7) |
| C(19)—F(6) | 1.383 (9) | 1.312 (7) |
| C(20)—F(7) | 1.288 (9) | 1.311 (11) |
| C(20)—F(8) | 1.376 (9) | 1.303 (10) |
| C(20)—F(9) | 1.323 (9) | 1.333 (12) |
| C(2)—F(10) | | 1.384 (6) |
| C(21)—F(11) | | 1.349 (7) |
| C(21)—F(12) | | 1.337 (6) |
| C(22)—F(13) | | 1.315 (8) |
| C(22)—F(14) | | 1.326 (8) |
| C(22)—F(15) | | 1.302 (8) |
| C(5)N(1)C(2) | 108.4 (4) | 110.0 (4) |
| N(1)C(2)C(3) | 110.0 (5) | 104.9 (4) |
| C(2)C(3)C(4) | 107.3 (5) | 101.4 (4) |
| C(3)C(4)C(5) | 106.0 (5) | 107.1 (4) |
| C(4)C(5)N(1) | 108.3 (5) | 111.8 (4) |
| N(1)C(2)C(18) | 119.5 (5) | 110.1 (5) |
| N(1)C(2)F(10) | | 110.6 (4) |
| C(3)C(2)C(18) | 130.5 (6) | 121.5 (5) |
| C(3)C(2)F(10) | | 107.1 (4) |
| C(18)C(2)F(10) | | 102.6 (4) |
| C(2)C(3)C(19) | 123.6 (6) | 112.9 (5) |
| C(2)C(3)C(21) | | 109.3 (4) |
| C(4)C(3)C(19) | 129.0 (6) | 105.2 (4) |
| C(4)C(3)C(21) | | 119.7 (4) |
| C(19)C(3)C(21) | | 108.3 (5) |
| C(5)C(4)C(2') | 115.8 (5) | 116.9 (5) |
| C(4)C(2')N(2) | 123.2 (5) | 122.9 (5) |
| C(2')N(2)C(6) | 119.9 (5) | 119.6 (4) |
| N(2)C(6)C(11) | 123.2 (5) | 123.2 (5) |
| C(6)C(11)C(5) | 115.2 (5) | 115.1 (5) |
| C(11)C(5)C(4) | 122.5 (5) | 122.5 (4) |
| C(4)C(2')C(20) | 124.8 (5) | 128.2 (5) |
| N(2)C(2')C(20) | 112.0 (5) | 108.8 (5) |
| C(11)C(6)C(7) | 121.2 (5) | 121.2 (5) |
| C(6)C(7)C(8) | 119.5 (6) | 120.2 (6) |
| C(7)C(8)C(9) | 119.7 (6) | 118.3 (6) |
| C(8)C(9)C(10) | 121.8 (6) | 122.4 (6) |
| C(9)C(10)C(11) | 120.9 (6) | 120.4 (5) |
| C(10)C(11)C(6) | 116.9 (5) | 117.4 (5) |
| C(2)N(1)C(12) | 127.1 (5) | 119.4 (4) |
| C(5)N(1)C(12) | 124.5 (5) | 122.7 (4) |

Table 4. *Torsional angles (°)*

| | Compound (1) | Compound (2) |
|--------------------|--------------|--------------|
| N(1)C(2)C(3)C(4) | 0.8 (7) | -21.5 (5) |
| C(2)C(3)C(4)C(5) | -1.1 (6) | 16.7 (5) |
| C(3)C(4)C(5)N(1) | 0.9 (6) | -6.0 (6) |
| C(2)N(1)C(5)C(4) | -0.4 (6) | -9.0 (6) |
| C(5)N(1)C(2)C(3) | -0.3 (7) | 19.4 (6) |
| C(5)N(1)C(2)C(18) | -179.1 (5) | 151.7 (5) |
| C(5)N(1)C(2)F(10) | | -95.7 (5) |
| N(1)C(2)C(3)C(19) | -174.7 (6) | 90.6 (6) |
| N(1)C(2)C(3)C(21) | | -148.8 (5) |
| C(12)N(1)C(2)C(3) | -179.4 (5) | 169.2 (5) |
| C(3)C(4)C(2')C(20) | -4.7 (11) | 10.9 (11) |
| C(5)C(4)C(2')C(20) | 174.1 (6) | -177.0 (6) |
| C(3)C(4)C(2')N(2) | 177.1 (6) | -171.6 (6) |
| C(3)C(4)C(5)C(11) | -177.2 (5) | 170.8 (5) |
| N(2)C(6)C(7)C(8) | -179.0 (6) | -179.5 (6) |
| N(2)C(6)C(11)C(10) | 178.8 (5) | 177.3 (6) |

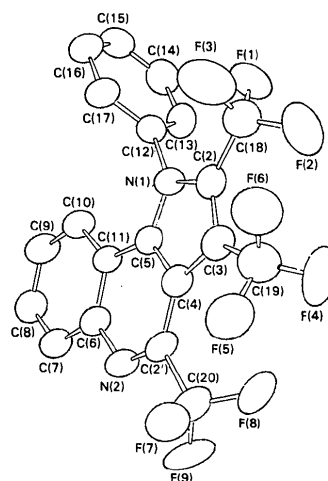


Fig. 1. ORTEP drawing (Johnson, 1976) of compound (1). Thermal ellipsoids are drawn at the 50% probability level.

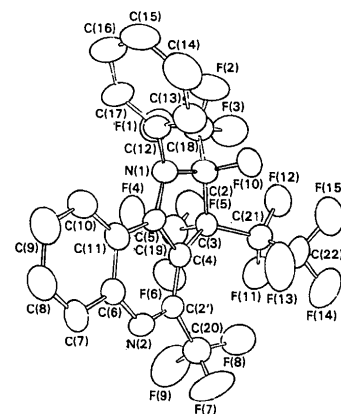


Fig. 2. ORTEP drawing (Johnson, 1976) of compound (2). Thermal ellipsoids are drawn at the 50% probability level.

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Structure and Atomic Charge Distribution of 4-Ethyl-3-methyl-1,2,3(4*H*)-benzotriazin-4-ol*

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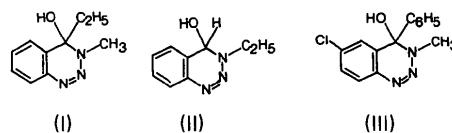
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Abstract. $C_{10}H_{13}N_3O$, $M_r = 191.23$, orthorhombic, *Pbca*, $a = 11.265$ (2), $b = 8.551$ (3), $c = 21.099$ (3) Å, $U = 2032.4$ Å³, $Z = 8$, $D_m = 1.25$ (1), $D_x = 1.250$ Mg m⁻³, Mo *K*α radiation, $\lambda = 0.71069$ Å, $\mu = 0.050$ mm⁻¹, $F(000) = 816$, $T = 293$ (3) K, final $R = 0.066$ for 760 observed reflections. The bicyclic system is relatively flat. As in similar 4-hydroxybenzotriazines, N(1) accepts a hydrogen bond from a nearby hydroxyl group. N(1) seems to be the preferred proton acceptor because of its accessibility as well as its negative charge, since N(1) bears only the third strongest negative charge in the molecule according to *ab initio* molecular-orbital calculations.

Introduction. The highly reactive N_3 unit in azides and triazines can be rendered more stable by incorporation into the bicyclic system of a 1,2,3-benzotriazine. Comparison of two 4-hydroxybenzotriazine structures (Lai, Schwalbe, Vaughan, Lafrance & Whiston, 1985) showed reasonable consistency of bond distances but up to 37° variation in three key torsion angles determining puckering of the triazine ring. Crystals of both molecules contained O–H...N intermolecular

hydrogen bonds in which N(1) was the proton acceptor. The structure of the title compound (I) was determined in order to ascertain whether the triazine ring was puckered as in 3-ethyl-1,2,3(4*H*)-benzotriazin-4-ol (II) or flat as in 6-chloro-3-methyl-4-phenyl-1,2,3-benzotriazin-4-ol (III), and to describe the mode of intermolecular hydrogen bonding. The hypothesis that the proton acceptor would be the most negative atom in the molecule was checked by performing *ab initio* molecular-orbital calculations.



Experimental. Needle crystal $1.04 \times 0.20 \times 0.16$ mm grown from aqueous solution. Density measured by flotation. Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo *K*α radiation. Unit-cell dimensions from least-squares analysis of setting angles from 25 reflections with $8.21 \leq \theta \leq 16.92^\circ$. Intensity data collected by the ω - 2θ technique for 1976 unique reflections measured in the range $2 \leq \theta \leq 25^\circ$, $0 \leq h \leq 13$, $-10 \leq k \leq 0$, $-25 \leq l \leq 0$. Three intensity and three orientation monitor reflections showed no significant variation during the experiment. 760 reflections

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