# Crystal structure of 5-fluoro-1-benzyluracil, $(C_6H_5)CH_2(C_4N_2H_2FO_2)$

J.-C. Ding[I], M.-C. Liu*[,I], L.-J. Zhang[II] and H.-Y. Wu[I]

[I] Wenzhou University, School of Chemistry and Materials Science, Wenzhou 325027, P. R. China
[II] Wenzhou University, Elementary Educational College, Wenzhou 325027, P. R. China

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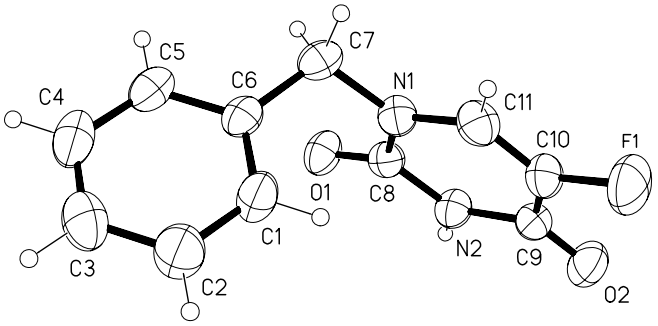


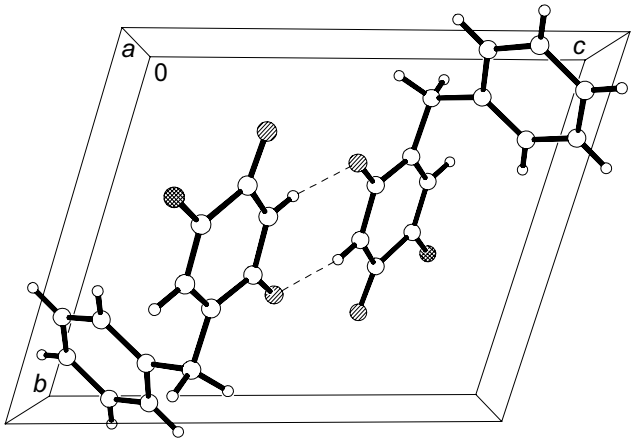


**Abstract**

$C_{11}H_9FN_2O_2$, triclinic, $P\bar{1}$ (no. 2), $a$ = 5.9762(9) Å, $b$ = 8.508(1) Å, $c$ = 10.316(2) Å, $\alpha$ = 106.634(3)°, $\beta$ = 93.496(3)°, $\gamma$ = 96.417(3)°, $V$ = 497.0 Å$^3$, $Z$ = 2, $R_{gt}(F)$ = 0.046, $wR_{ref}(F^2)$ = 0.102, $T$ = 298 K.

**Source of material**

Benzyl bromide (30 mmol, 5.13 g) in water (10 ml) was added to a solution of 5-fluoro-uracil (30 mmol, 3.89 g), potassium hydroxide (11.5 mmol, 6.41 g) and water (20 ml) at 313 K. The mixture was cooled after 2 h reaction and the pH was adjusted to 5.5 using hydrochloric acid. The precipitate was filtered off and the pH of the filtrate was adjusted to 1, again using hydrochloric acid. The solution was cooled for 6 h in a refrigerator and then filtered. The precipitate was washed with water and dried to obtain the title compound. Single crystals were obtained by recrystallization from anhydrous ethanol.

**Discussion**

5-Fluorouracil (5FU) is an antimetabolite with good antimicrobial and antitumor activity, but its administration is accompanied by significant toxic side effects and delivery problem [1-4]. In order to improve the topical delivery of 5FU and reduce the side effects, many derivatives of 5FU have been synthesized, some of which have better biological activity [5-7]. However, to our knowledge, the crystal structure of 5-fluoro-1-benzyluracil has not been reported so far. In order to better understand the benzylation of uracil, the compound was synthesized and its crystal structure has been determined.

The title molecule consists of two different rings. In the crystal structure, the phenyl ring (C1/C2/C3/C4/C5/C6) and the heterocyclic ring (C8/C9/C10/C11/N1/N2) are each almost planar (figure, top). The dihedral angle between their best planes is 85.64(6)°. Moreover, the four C—N bond lengths in the uracil ring range from 1.367(2) Å to 1.386(2) Å, which is shorter than a C—N single bond length (ca 1.443 Å [8], but longer than a typical C=N bond length (ca 1.269 Å), indicating the electron delocalization. Furthermore, the C6—C7 and C7—N1, distances of 1.504(3) Å and 1.475(2) Å, respectively, clearly indicate two single bonds. Therefore, there is no conjugation between the phenyl ring and the heterocyclic ring. One N–H···O hydrogen bonds occur in the crystal structure. Two adjacent molecules are pairwise linked by the intermolecular N2–H2···O1(1–$x$,2–$y$,–$z$) hydrogen bonds to form a dimer structure (figure, bottom).

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | colorless block, size 0.12 × 0.24 × 0.26 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 1.16 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker SMART APEX CCD, $\varphi/\omega$ |
| $2\theta_{max}$: | 50.44° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 2673, 1772 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 1420 |
| $N(param)_{refined}$: | 145 |
| Program: | SHELXTL [9] |

**Table 2.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(2) | 2*i* | 0.3458 | 1.0874 | 0.0696 | 0.044 |
| H(1) | 2*i* | 0.3309 | 0.8322 | 0.4167 | 0.055 |
| H(2A) | 2*i* | 0.6144 | 0.8338 | 0.5778 | 0.067 |
| H(3) | 2*i* | 0.8516 | 0.6324 | 0.5337 | 0.068 |
| H(4) | 2*i* | 0.8046 | 0.4313 | 0.3264 | 0.064 |
| H(5) | 2*i* | 0.5233 | 0.4314 | 0.1632 | 0.051 |
| H(7A) | 2*i* | 0.2462 | 0.5654 | 0.0785 | 0.049 |
| H(7B) | 2*i* | 0.0712 | 0.5648 | 0.1856 | 0.049 |
| H(11) | 2*i* | –0.1173 | 0.7977 | 0.2663 | 0.050 |

* Correspondence author (e-mail: mcl@wznc.zj.cn)

**Table 3.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | *x* | *y* | *z* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| F(1) | 2*i* | –0.2351(2) | 1.0758(2) | 0.2882(1) | 0.0525(8) | 0.0620(8) | 0.088(1) | 0.0211(6) | 0.0324(7) | 0.0204(7) |
| O(1) | 2*i* | 0.4609(2) | 0.8176(2) | 0.0460(1) | 0.0454(8) | 0.0404(8) | 0.0524(9) | 0.0133(6) | 0.0189(7) | 0.0190(6) |
| O(2) | 2*i* | 0.0490(2) | 1.2472(2) | 0.1584(2) | 0.056(1) | 0.0386(8) | 0.068(1) | 0.0138(7) | 0.0089(7) | 0.0170(7) |
| N(1) | 2*i* | 0.1583(3) | 0.7908(2) | 0.1644(2) | 0.0352(9) | 0.0369(9) | 0.0396(9) | 0.0049(7) | 0.0059(7) | 0.0161(7) |
| N(2) | 2*i* | 0.2547(3) | 1.0319(2) | 0.1069(2) | 0.0385(9) | 0.0340(9) | 0.0412(9) | 0.0057(7) | 0.0084(7) | 0.0164(7) |
| C(1) | 2*i* | 0.4266(4) | 0.7512(2) | 0.3975(2) | 0.053(1) | 0.042(1) | 0.045(1) | 0.020(1) | 0.007(1) | 0.0120(9) |
| C(2) | 2*i* | 0.5958(4) | 0.7521(3) | 0.4944(2) | 0.066(2) | 0.058(1) | 0.043(1) | 0.018(1) | 0.001(1) | 0.012(1) |
| C(3) | 2*i* | 0.7376(4) | 0.6323(3) | 0.4682(2) | 0.053(1) | 0.066(2) | 0.059(2) | 0.014(1) | 0.001(1) | 0.031(1) |
| C(4) | 2*i* | 0.7095(4) | 0.5127(3) | 0.3446(2) | 0.048(1) | 0.049(1) | 0.075(2) | 0.019(1) | 0.018(1) | 0.031(1) |
| C(5) | 2*i* | 0.5405(3) | 0.5126(2) | 0.2469(2) | 0.050(1) | 0.032(1) | 0.049(1) | 0.0073(9) | 0.016(1) | 0.0130(9) |
| C(6) | 2*i* | 0.3962(3) | 0.6327(2) | 0.2727(2) | 0.039(1) | 0.030(1) | 0.039(1) | 0.0036(8) | 0.0091(8) | 0.0156(8) |
| C(7) | 2*i* | 0.2069(3) | 0.6256(2) | 0.1673(2) | 0.048(1) | 0.031(1) | 0.045(1) | 0.0018(9) | 0.0076(9) | 0.0131(8) |
| C(8) | 2*i* | 0.3017(3) | 0.8761(2) | 0.1021(2) | 0.036(1) | 0.034(1) | 0.032(1) | 0.0038(8) | 0.0009(8) | 0.0110(8) |
| C(9) | 2*i* | 0.0781(3) | 1.1105(2) | 0.1646(2) | 0.037(1) | 0.036(1) | 0.038(1) | 0.0071(9) | –0.0021(8) | 0.0069(8) |
| C(10) | 2*i* | –0.0592(3) | 1.0101(2) | 0.2283(2) | 0.034(1) | 0.045(1) | 0.045(1) | 0.0105(9) | 0.0091(9) | 0.0098(9) |
| C(11) | 2*i* | –0.0211(3) | 0.8590(2) | 0.2258(2) | 0.035(1) | 0.049(1) | 0.045(1) | 0.0035(9) | 0.0089(9) | 0.0181(9) |

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