*ORTEP*, Johnson, 1965). The molecules are piled up along the *c* axis and form a parallel plane-to-plane stack.

IR, NMR, mass spectrum and elemental analysis data concerning compound (6) could fit both structures (5) and (6). Our X-ray analysis allows us to differentiate clearly between these two possibilities and to assign the correct structure of this molecule. This result now calls for an interpretation of this unexpected transformation and offers new perspectives for the synthesis of potential anticancer drugs.

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# Structure of 6-Amino-1,3-dimethyl-5-[5-(dimethylamino)-1,2,3,4-tetrazol-1-yl]uracil Hemihydrate: $C_9H_{14}N_8O_2.\frac{1}{2}H_2O$

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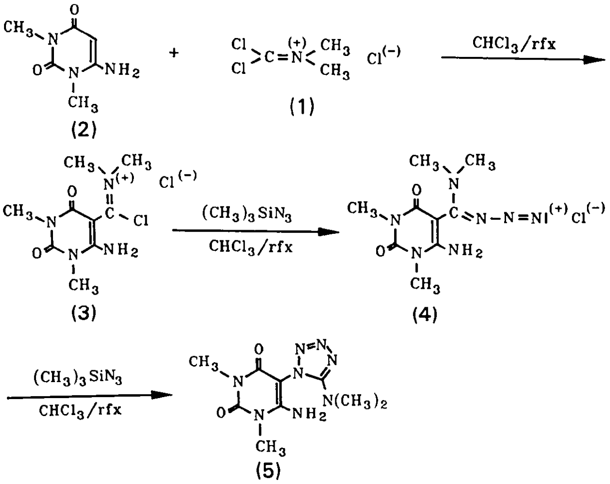
**Abstract.** $M_r = 550.55$, orthorhombic, $P2_1ca$, $a = 14.573$ (5), $b = 7.594$ (3), $c = 24.740$ (6) Å, $V = 2738$ (8) Å$^3$, $Z = 8$, $D_x = 1.336$ Mg m$^{-3}$, $\lambda$(Cu $K\alpha$) = 1.5418 Å, $\mu = 0.10$ mm$^{-1}$, $F(000) = 1160$, $T = 293$ K. The structure was solved by direct methods and refined to $R = 0.057$ and $wR = 0.058$ for 2269 independent reflections. The title compound was obtained starting from 1,3-dimethyl-6-aminouracil by condensation with phosgeniminium chloride followed by reaction with trimethylsilyl azide.

**Introduction.** While attempting to prepare new heterocyclic compounds of biological interest starting from uracils, we found that phosgeniminium chloride [*N*,*N*-dimethyldichloromethyleniminium chloride; (1)], condenses quantitatively with 1,3-dimethyl-6-aminouracil (2) affording the thermally stable 1,3-dimethyl-4-amino-5-(chloromethine-*N*,*N*-dimethyliminium)uracil (3) (Kokel, Lespagnol & Viehe, 1980) (see scheme below).



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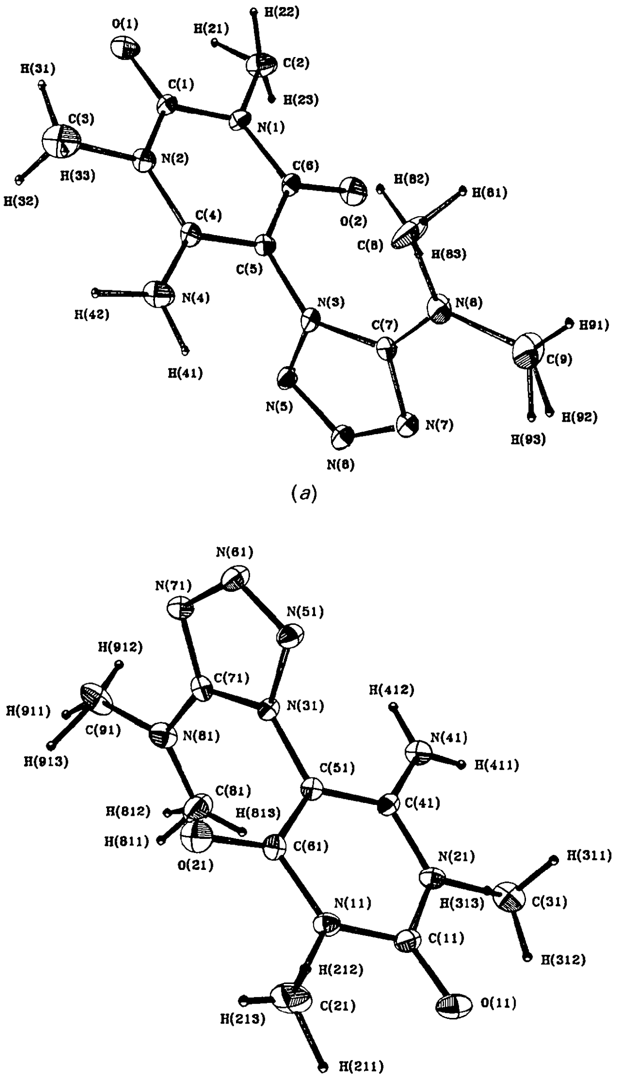
Compound (3) is a powerful electrophilic synthon. It reacts readily with nucleophiles to give various heterocyclic systems containing a uracilic moiety (Kokel, Lespagnol & Viehe, 1980; Kokel, Royer, Declercq, Germain & Van Meerssche, 1981; Kokel, Guillaumel & Royer, 1983; Kokel, 1987; Kokel, Hubert-Habart, Cousson & Bachet, 1990). Thus upon reaction with two equivalents of trimethylsilyl azide, it gives a very good yield of 6-amino-1,3-dimethyl-5-[5-(dimethylamino)-1,2,3,4-tetrazol-1-yl]uracil (5) *via* formation of the amidazide (4) which was not isolated.

**Experimental.** The tetrazole (5) was synthesized as described previously (Kokel, 1987) and recrystallized twice from water. Large crystals could thus be formed and selected. A single crystal, 0.20 × 0.20 × 0.35 mm, was selected for data collection. The unit-cell dimensions and reflection intensities were measured using a Philips PW1100 four-circle diffractometer and graphite-monochromatized radiation [$\lambda$(Cu $K\alpha$) = 1.5418 Å], scan type 'flying step scan', scan range 1.5° $\theta$, scan speed 0.025° $\theta\ \mathrm{s}^{-1}$, $\theta$ range 1–68°, $0 \le h \le 16$, $0 \le k \le 9$, $0 \le l \le 29$, 2891 data collected, three standard reflections (620, 0,0,12 and $\bar{6}\bar{2}0$), decomposition less than 0.02, absorption correction using *DIFABS* (Walker & Stuart, 1983). Structure solved by direct methods [*SHELXS*86 and *CRYSTALS* from Sheldrick (1986) and Watkin, Carruthers & Betteridge (1985) respectively], final $R = 0.057$ and $wR = 0.058$ for 2269 independent reflections such that $I \ge 3\sigma(I)$, $w = 1.0$ ($S = 5.8$, $\Delta/\sigma_{\max} = 0.12$), form factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–101), on the last Fourier difference map $\rho_{\max} = 0.02$, $\rho_{\min} = -0.02\ \mathrm{e\ Å^{-3}}$. Computer used: VAX 6310.

**Discussion.** There are two independent molecules and a water molecule in the asymmetric unit. A view of these two molecules (*A* and *B*) showing their conformation and the numbering system is presented in Fig. 1. Atomic positional parameters are reported in Table 1,* intramolecular bond distances and angles are given in Table 2. The two molecules are very similar: the angles between the two rings are 90.73° for molecule *A* and 92.61° for molecule *B*, and the bond-length differences, >0.03 Å, concern the methyl groups belonging to the dimethylamino substituent of the tetrazolo ring. The crystal structure shown in Fig. 2 is viewed along the *b* axis (figures were drawn using *ORTEP*, Johnson, 1965). The six-membered and five-membered rings of *A* and *B* are parallel (ring angles of 2.05 and 2.15° respectively). In addition the six-membered rings are in front of each other in reverse positions and the five-membered rings protrude in opposite directions. The molecules are piled up approximately along the *c* axis and the water molecule is bound to two *A* molecules and four *B* molecules.

The X-ray results prove the structure of compound (5), whose IR and NMR data were insufficient to determine its detailed structure. It henceforth opens the way to the synthesis of new tetrazolo derivatives of pharmaceutical interest.



(*a*)

(*b*)

Fig. 1. Molecular structure of $C_9H_{14}N_8O_2.\frac{1}{2}H_2O$. (*a*) Molecule *A*. (*b*) Molecule *B*.

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* Lists of structure factors, anisotropic thermal parameters, and intramolecular bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54153 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic positional parameters with* $U_{eq}$ *values for non-*H *atoms*

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| C(1) | 0.5825 (5) | 1.1664 (9) | 0.0903 (3) | 0.0391 |
| C(2) | 0.7414 (6) | 1.666 (1) | 0.1184 (3) | 0.0615 |
| C(3) | 0.4204 (6) | 1.555 (1) | 0.0681 (4) | 0.0596 |
| C(4) | 0.5360 (5) | 1.0128 (9) | 0.0088 (3) | 0.0354 |
| C(5) | 0.6257 (5) | 0.9645 (9) | −0.0005 (3) | 0.0337 |
| C(6) | 0.6961 (5) | 1.0069 (9) | 0.0361 (3) | 0.0381 |
| C(7) | 0.6698 (5) | 0.7138 (8) | −0.0633 (3) | 0.0363 |
| C(8) | 0.605 (1) | 0.563 (1) | 0.0151 (4) | 0.0671 |
| C(9) | 0.685 (1) | 0.403 (1) | −0.0552 (4) | 0.0716 |
| C(11) | 0.5782 (6) | 0.330 (1) | −0.3336 (3) | 0.0427 |
| C(21) | 0.4197 (7) | 0.346 (1) | −0.3607 (4) | 0.0664 |
| C(31) | 0.7415 (6) | 0.325 (1) | −0.3117 (3) | 0.0548 |
| C(41) | 0.6304 (5) | 0.4696 (8) | −0.2508 (3) | 0.0348 |
| C(51) | 0.5419 (5) | 0.5223 (8) | −0.2406 (3) | 0.0325 |
| C(61) | 0.4693 (6) | 0.4872 (9) | −0.2761 (3) | 0.0391 |
| C(71) | 0.5030 (5) | 0.7698 (8) | −0.1750 (3) | 0.0363 |
| C(81) | 0.5657 (8) | 0.922 (1) | −0.2539 (4) | 0.0752 |
| C(91) | 0.4873 (8) | 1.086 (1) | −0.1786 (4) | 0.0639 |
| N(1) | 0.6719 (4) | 1.1129 (8) | 0.0799 (2) | 0.0408 |
| N(2) | 0.5158 (4) | 1.1075 (8) | 0.0550 (2) | 0.0373 |
| N(3) | 0.6500 (4) | 0.8823 (7) | −0.0488 (2) | 0.0364 |
| N(4) | 0.4667 (5) | 0.974 (1) | −0.0247 (3) | 0.0484 |
| N(5) | 0.6765 (5) | 0.9895 (7) | −0.0923 (2) | 0.0445 |
| N(6) | 0.7054 (4) | 0.8851 (8) | −0.1296 (2) | 0.0440 |
| N(7) | 0.7031 (4) | 0.7126 (7) | −0.1138 (2) | 0.0416 |
| N(8) | 0.6585 (6) | 0.5692 (8) | −0.0334 (3) | 0.0543 |
| N(11) | 0.4921 (4) | 0.3888 (8) | −0.3217 (2) | 0.0413 |
| N(21) | 0.6480 (4) | 0.3809 (7) | −0.2978 (2) | 0.0377 |
| N(31) | 0.5196 (4) | 0.6023 (7) | −0.1901 (2) | 0.0316 |
| N(41) | 0.6983 (5) | 0.5014 (8) | −0.2166 (2) | 0.0425 |
| N(51) | 0.4973 (5) | 0.4940 (7) | −0.1470 (2) | 0.0444 |
| N(61) | 0.4700 (5) | 0.5963 (8) | −0.1100 (2) | 0.0509 |
| N(71) | 0.4727 (4) | 0.7687 (7) | −0.1239 (2) | 0.0404 |
| N(81) | 0.5108 (5) | 0.9136 (8) | −0.2042 (3) | 0.0520 |
| O(1) | 0.5644 (4) | 1.2584 (8) | 0.1291 (2) | 0.0522 |
| O(2) | 0.7779 (4) | 0.9619 (8) | 0.0303 (2) | 0.0546 |
| O(11) | 0.5946 (5) | 0.2388 (8) | −0.3721 (2) | 0.0532 |
| O(21) | 0.3894 (4) | 0.5383 (8) | −0.2688 (2) | 0.0543 |
| O(100) ($H_2O$) | 0.3157 (6) | −0.054 (1) | −0.2711 (3) | 0.1156 |

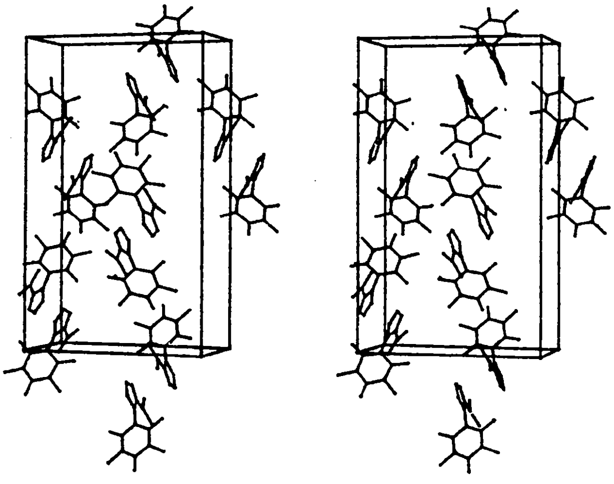

Fig. 2. Stereoscopic view of $C_9H_{14}N_8O_2.\frac{1}{2}H_2O$.

Financial support from the 'Institut Curie' (Contrat coopératif, décision 90-24) is gratefully acknowledged.

Table 2. *Intramolecular bond distances* (Å) *and angles* (°)

| Molecule *A* | | Molecule *B* | |
|---|---|---|---|
| C(1)—N(1) | 1.39 (1) | C(11)—N(11) | 1.364 (9) |
| C(1)—N(2) | 1.381 (9) | C(11)—N(21) | 1.404 (9) |
| C(1)—O(1) | 1.215 (8) | C(11)—O(11) | 1.201 (8) |
| C(2)—N(1) | 1.449 (9) | C(21)—N(11) | 1.47 (1) |
| C(3)—N(2) | 1.47 (1) | C(31)—N(21) | 1.47 (1) |
| C(4)—C(5) | 1.38 (1) | C(41)—C(51) | 1.37 (1) |
| C(4)—N(2) | 1.381 (9) | C(41)—N(21) | 1.368 (9) |
| C(4)—N(4) | 1.34 (1) | C(41)—N(41) | 1.32 (1) |
| C(5)—C(6) | 1.40 (1) | C(51)—C(61) | 1.40 (1) |
| C(5)—N(3) | 1.396 (9) | C(51)—N(31) | 1.428 (8) |
| C(6)—N(1) | 1.395 (9) | C(61)—N(11) | 1.394 (9) |
| C(6)—O(2) | 1.25 (1) | C(61)—O(21) | 1.24 (1) |
| C(7)—N(3) | 1.359 (8) | C(71)—N(31) | 1.348 (8) |
| C(7)—N(7) | 1.340 (8) | C(71)—N(71) | 1.337 (8) |
| C(7)—N(8) | 1.335 (9) | C(71)—N(81) | 1.314 (9) |
| C(8)—N(8) | 1.43 (1) | C(81)—N(81) | 1.47 (1) |
| C(9)—N(8) | 1.43 (1) | C(91)—N(81) | 1.50 (1) |
| N(3)—N(5) | 1.403 (8) | N(31)—N(51) | 1.386 (7) |
| N(5)—N(6) | 1.287 (8) | N(51)—N(61) | 1.264 (8) |
| N(6)—N(7) | 1.367 (8) | N(61)—N(71) | 1.354 (8) |
| | | | |
| N(2)—C(1)—N(1) | 116.6 (6) | N(21)—C(11)—N(11) | 116.1 (6) |
| O(1)—C(1)—N(1) | 121.2 (7) | O(11)—C(11)—N(11) | 122.9 (7) |
| O(1)—C(1)—N(2) | 122.2 (7) | O(11)—C(11)—N(21) | 121.0 (8) |
| N(2)—C(4)—C(5) | 118.6 (6) | N(21)—C(41)—C(51) | 118.3 (7) |
| N(4)—C(4)—C(5) | 123.7 (6) | N(41)—C(41)—C(51) | 122.1 (6) |
| N(4)—C(4)—N(2) | 117.8 (6) | N(41)—C(41)—N(21) | 119.5 (7) |
| C(6)—C(5)—C(4) | 121.7 (6) | C(61)—C(51)—C(41) | 122.7 (6) |
| N(3)—C(5)—C(4) | 120.2 (6) | N(31)—C(51)—C(41) | 119.8 (6) |
| N(3)—C(5)—C(6) | 118.0 (6) | N(31)—C(51)—C(61) | 117.3 (6) |
| N(1)—C(6)—C(5) | 116.6 (7) | N(11)—C(61)—C(51) | 115.5 (7) |
| O(2)—C(6)—C(5) | 124.1 (7) | O(21)—C(61)—C(51) | 124.0 (7) |
| O(2)—C(6)—N(1) | 119.2 (7) | O(21)—C(61)—N(11) | 120.6 (7) |
| N(7)—C(7)—N(3) | 109.2 (6) | N(71)—C(71)—N(31) | 108.4 (6) |
| N(8)—C(7)—N(3) | 127.0 (6) | N(81)—C(71)—N(31) | 128.0 (6) |
| N(8)—C(7)—N(7) | 123.8 (6) | N(81)—C(71)—N(71) | 123.6 (6) |
| C(2)—N(1)—C(1) | 116.8 (6) | C(21)—N(11)—C(11) | 116.6 (6) |
| C(6)—N(1)—C(1) | 123.4 (6) | C(61)—N(11)—C(11) | 124.7 (6) |
| C(6)—N(1)—C(2) | 119.8 (7) | C(61)—N(11)—C(21) | 118.7 (7) |
| C(3)—N(2)—C(1) | 116.4 (6) | C(31)—N(21)—C(11) | 116.4 (6) |
| C(4)—N(2)—C(1) | 122.8 (6) | C(41)—N(21)—C(11) | 122.5 (6) |
| C(4)—N(2)—C(3) | 120.7 (6) | C(41)—N(21)—C(31) | 121.0 (6) |
| C(7)—N(3)—C(5) | 134.5 (6) | C(71)—N(31)—C(51) | 133.3 (6) |
| N(5)—N(3)—C(5) | 117.9 (5) | N(51)—N(31)—C(51) | 118.4 (5) |
| N(5)—N(3)—C(7) | 106.6 (5) | N(51)—N(31)—C(71) | 107.7 (5) |
| N(6)—N(5)—N(3) | 106.4 (5) | N(61)—N(51)—N(31) | 105.4 (5) |
| N(7)—N(6)—N(5) | 112.2 (6) | N(71)—N(61)—N(51) | 113.6 (6) |
| N(6)—N(7)—C(7) | 105.6 (5) | N(61)—N(71)—C(71) | 104.8 (5) |
| C(8)—N(8)—C(7) | 124.0 (6) | C(81)—N(81)—C(71) | 123.0 (7) |
| C(9)—N(8)—C(7) | 119.0 (6) | C(91)—N(81)—C(71) | 118.4 (7) |
| C(9)—N(8)—C(8) | 115.8 (7) | C(91)—N(81)—C(81) | 116.2 (7) |

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