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Structure of 5,7-Dimethyl-2-(dimethylamino)oxazolo[5,4-*d*]pyrimidine-4,6(5*H*,7*H*)-dione: $C_9H_{12}N_4O_3$

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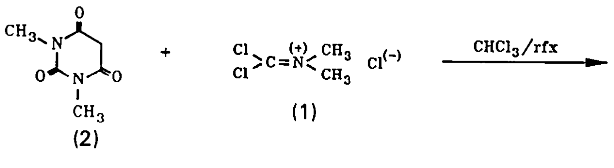
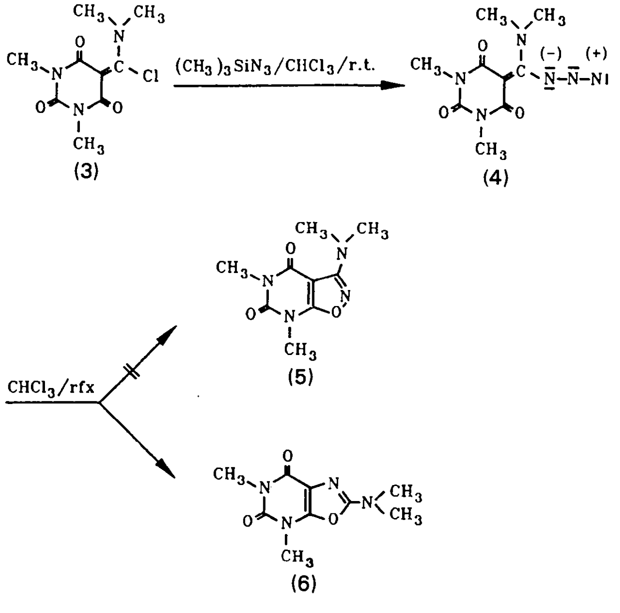
Abstract. $M_r = 224.22$, monoclinic, $P2_1/n$, $a = 16.732\,(6)$, $b = 8.406\,(4)$, $c = 7.375\,(3)$ Å, $\beta = 91.68\,(3)°$, $V = 1037\,(6)$ Å^3, $Z = 4$, $D_x = 1.436$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 0.10$ mm^{-1}, $F(000) = 472$, $T = 293$ K. The structure was solved by direct methods and refined to $R = 0.055$ and $wR = 0.051$ for 1650 independent reflections. The title compound is one of a series of novel oxazolo[5,4-*d*]pyrimidines, obtained starting from the corresponding barbituric acids, *via* condensation with phosgeniminium chloride and subsequent reaction with trimethylsilyl azide.

Introduction. While focusing on uracils as starting materials for the preparation of new heterocyclic compounds of biological interest, we found that the condensation of phosgeniminium chloride [*N*,*N*-dimethyldichloromethyleniminium chloride; (1)], with 1,3-dimethylbarbituric acid (2) affords the thermally stable 1,3-dimethyl-5-(*N*,*N*-dimethylaminochloromethylene)barbituric acid (3) (see scheme below) in quantitative yield (Kokel, Lespagnol & Viehe, 1980).

Chloroenamine (3) reacts readily with trimethylsilyl azide in chloroform to give the unstable amidazide (4) which is thermally converted in very good yield to the unexpected 5,7-dimethyl-2-(dimethylamino)oxazolo[5,4-*d*]pyrimidine-4,6(5*H*,7*H*)-dione (6) (Kokel, Hubert-Habart, Cousson & Bachet, 1990), described, in previous reports, as its isomeric 5,7-dimethyl-3-(dimethylamino)isoxazolo[5,4-*d*]pyrimidine-4,6(5*H*,7*H*)-dione (5) (Kokel, Lespagnol & Viehe, 1980; Kokel, 1987).





Experimental. The molecule (6) was obtained as described earlier (Kokel, Lespagnol & Viehe, 1980) and recrystallized twice from isopropanol. Large crystals could be thus formed and selected. A single crystal, 0.20 × 0.30 × 0.30 mm, was selected for data collection. The unit-cell dimensions and reflection intensities were measured with a Philips PW1100 four-circle diffractometer, graphite-monochroma-

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

	x	y	z	U_{eq}
C(1)	0.8979 (2)	0.1476 (4)	0.8316 (4)	0.0416
C(2)	0.9634 (2)	−0.0492 (3)	0.7344 (4)	0.0399
C(3)	0.9903 (2)	−0.1963 (3)	0.6634 (4)	0.0439
C(4)	1.1288 (2)	−0.0801 (4)	0.6942 (4)	0.0440
C(5)	1.0162 (2)	0.0640 (3)	0.7774 (3)	0.0369
C(6)	1.1097 (3)	−0.3482 (5)	0.5781 (5)	0.0618
C(7)	1.1478 (2)	0.1932 (5)	0.8145 (6)	0.0591
C(8)	0.7604 (2)	0.2161 (6)	0.8644 (6)	0.0718
C(9)	0.8692 (3)	0.4168 (5)	0.9330 (5)	0.0644
N(1)	0.8451 (2)	0.2546 (3)	0.8879 (4)	0.0559
N(2)	0.8855 (1)	0.0049 (3)	0.7708 (3)	0.0458
N(3)	1.0747 (1)	−0.2005 (3)	0.6497 (3)	0.0429
N(4)	1.0962 (1)	0.0587 (3)	0.7620 (3)	0.0426
O(1)	0.9783 (1)	0.1955 (2)	0.8410 (2)	0.0421
O(2)	0.9493 (1)	−0.3102 (3)	0.6168 (3)	0.0661
O(3)	1.2003 (1)	−0.0950 (3)	0.6766 (3)	0.0608

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

C(1)—N(1)	1.335 (4)	C(4)—N(4)	1.387 (4)
C(1)—N(2)	1.295 (4)	C(4)—O(3)	1.214 (3)
C(1)—O(1)	1.404 (3)	C(5)—N(4)	1.348 (3)
C(2)—C(3)	1.421 (4)	C(5)—O(1)	1.365 (3)
C(2)—C(5)	1.331 (4)	C(6)—N(3)	1.477 (4)
C(2)—N(2)	1.412 (3)	C(7)—N(4)	1.467 (4)
C(3)—N(3)	1.420 (4)	C(8)—N(1)	1.459 (5)
C(3)—O(2)	1.220 (3)	C(9)—N(1)	1.457 (5)
C(4)—N(3)	1.390 (4)		

N(2)—C(1)—N(1)	129.2 (3)	O(1)—C(5)—C(2)	110.4 (2)
O(1)—C(1)—N(1)	115.6 (3)	O(1)—C(5)—N(4)	122.0 (2)
O(1)—C(1)—N(2)	115.2 (2)	C(8)—N(1)—C(1)	117.5 (3)
C(5)—C(2)—C(3)	119.6 (3)	C(9)—N(1)—C(1)	121.4 (3)
N(2)—C(2)—C(3)	130.8 (3)	C(9)—N(1)—C(8)	119.8 (3)
N(2)—C(2)—C(5)	109.5 (2)	C(2)—N(2)—C(1)	103.0 (2)
N(3)—C(3)—C(2)	112.0 (2)	C(4)—N(3)—C(3)	127.4 (2)
O(2)—C(3)—C(2)	127.2 (3)	C(6)—N(3)—C(3)	116.8 (3)
O(2)—C(3)—N(3)	120.8 (3)	C(6)—N(3)—C(4)	115.8 (3)
N(4)—C(4)—N(3)	116.0 (2)	C(5)—N(4)—C(4)	117.3 (2)
O(3)—C(4)—N(3)	122.5 (3)	C(7)—N(4)—C(4)	120.6 (2)
O(3)—C(4)—N(4)	121.5 (3)	C(7)—N(4)—C(5)	122.0 (3)
N(4)—C(5)—C(2)	127.7 (3)	C(5)—O(1)—C(1)	101.9 (2)

tized radiation [$\lambda(\text{Cu } K\alpha) = 1.5418$ Å], scan type 'flying step scan', scan range 1.8°θ, scan speed 0.02°θ s^{-1}, θ range 1–67°, $-20 \le h \le 20$, $0 \le k \le 10$, $0 \le l \le 8$, 2183 data collected, 2037 unique, three standard reflections ($\bar{5}31$, 800 and $53\bar{1}$), decomposition less than 0.01, absorption correction using *DIFABS* (Walker & Stuart, 1983), minimum absorption correction 0.424, maximum 1.593. Structure solved by direct methods [*SHELXS*86 and *CRYSTALS* from Sheldrick (1986) and Watkin, Carruthers & Betteridge (1985) respectively], final $R = 0.055$ and $wR = 0.051$ for 1650 independent reflections such that $I \ge 3\sigma(I)$, $w = 1.0$ ($S = 2.6$, $\Delta/\sigma_{\max} = 0.12$), form factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–101), on the last Fourier difference map $\rho_{\max} = 0.1$, $\rho_{\min} = -0.2$ e Å^{-3}. Computer used: VAX 6310.

Discussion. A view of the molecule showing its 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 crystal structure shown in Fig. 2 is viewed along the *a* axis (figures drawn using

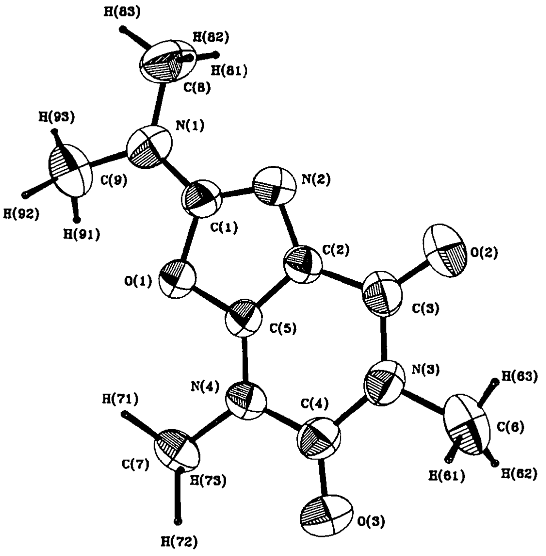


Fig. 1. Molecular structure of $C_9H_{12}N_4O_3$.

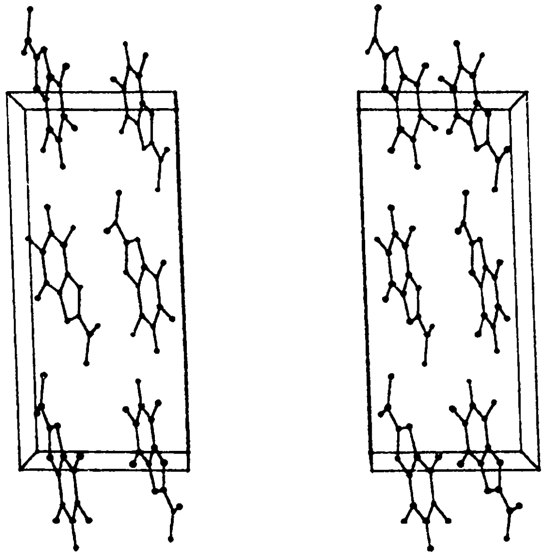


Fig. 2 Stereoscopic view of $C_9H_{12}N_4O_3$.

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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 54152 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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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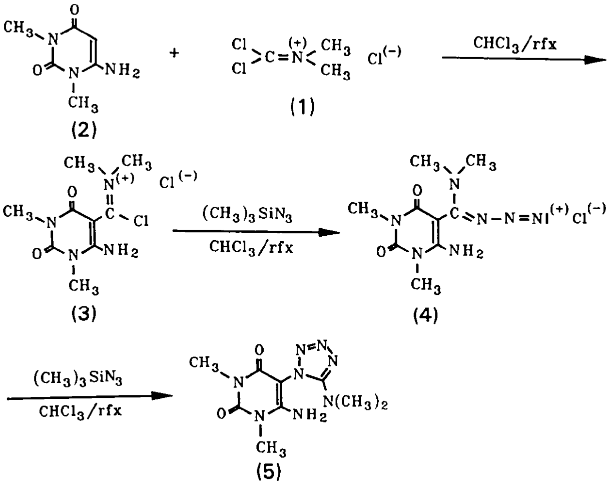
Abstract. $M_r = 550.55$, orthorhombic, $P2_1ca$, $a = 14.573\,(5)$, $b = 7.594\,(3)$, $c = 24.740\,(6)$ Å, $V = 2738\,(8)$ Å^3, $Z = 8$, $D_x = 1.336$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1.5418$ Å, $\mu = 0.10$ mm^{-1}, $F(000) = 1160$, $T = 293$ K. The structure was solved by direct methods 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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