Related literature. Preparation of similar compounds, 4-nitroisoxazoline *N*-oxides, have been reported by Gabitov, Kremleva & Fridman (1978), and a heterocycle with the same ring connectivity has been reported by Delugeard, Baudour & Messager (1974).

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Structure of an Azapyrimidine Derivative

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Abstract. 5-Aza-6-methoxyuracil (1,2,3,4-tetrahydro-6-methoxy-1,3,5-triazine-2,4-dione), $C_4H_7N_3O_3$, M_r = 145·12, monoclinic, $P2_1/c$, a = 9·994 (5), b = 4·316 (1), c = 15·891 (8) Å, β = 115·40 (3)°, V = 619·1 (4) Å^3, Z = 4, D_x = 1·557 Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ = 1·11 mm^{-1}, $F(000)$ = 304, T = 295 K, final R = 0·050, wR = 0·058 for 756 independent observed reflections. The azapyrimidine ring is in a shallow boat configuration, with the methoxy axial to the N(1)–C(2)–N(5) plane of the boat. The puckering parameter, the angle between the base plane N(1)–C(2)–C(4)–N(5) and the N(1)–C(6)–N(5) and C(2)–N(3)–C(4) planes, is 30·0 (3) and 10·7 (3)° respectively. Each of the three ring N atoms participates in an intermolecular hydrogen bond; N(1) acts as a donor to O(6)′($1-x$, $y-0{\cdot}5$, $0{\cdot}5-z$), N(3) is a donor to O(2)′($1-x$, $-y$, $-z$), and N(5) is a donor to O(4)′($-x$, $y-0{\cdot}5$, $0{\cdot}5-z$), with N···O′ distances of 2·905 (4), 2·853 (4), and 2·817 (3) Å respectively.

Experimental. A clear, colorless 0·12 × 0·20 × 0·30 mm data crystal was provided by C. Coon of Lawrence Livermore Laboratory. Automated Nicolet *R*3*m* diffractometer with incident-beam graphite monochromator, 25 centered reflections within $40 \le 2\theta \le 77°$ used for determining lattice parameters. $(\mathrm{Sin}\theta/\lambda)_{max}$ = 0·55 Å^{-1}, range of *hkl*: $0 \le h \le 10$, $0 \le k \le 4$, $-16 \le l \le 15$. Standards 300, 024, 004, monitored every 60 reflections with random variation of 3·1% over data collection, $\theta/2\theta$ mode, scan width (2·0 + $\Delta_{\alpha1\alpha2}$)°, scan rate a function of count rate (2·0° min^{-1} minimum, 30° min^{-1} maximum), 1102 reflections measured, 846 unique, R_{int} = 0·008, 756 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz and polarization, but not absorption effects. Structure solved by direct methods. The least-squares refinement

Table 1. *Atom coordinates* (×10^4) *and temperature parameters* (Å^2 ×10^3)

	x	y	z	U_{eq}
N(1)	4386 (3)	−45 (6)	3215 (2)	40 (1)*
C(2)	4640 (3)	−1972 (7)	3937 (2)	35 (1)*
N(3)	3399 (2)	−3238 (6)	3956 (2)	37 (1)*
C(4)	1972 (3)	−2294 (7)	3415 (2)	37 (1)*
N(5)	1834 (3)	−335 (6)	2712 (2)	40 (1)*
C(6)	2969 (3)	94 (7)	2417 (2)	38 (1)*
O(2)	5891 (2)	−2519 (5)	4535 (1)	45 (1)*
O(4)	938 (2)	−3139 (6)	3581 (1)	51 (1)*
O(6)	2967 (2)	−2267 (5)	1786 (1)	42 (1)*
C(7)	1712 (3)	−2099 (9)	884 (2)	54 (1)*
H(1)	5092 (37)	724 (74)	3166 (21)	48
H(3)	3524 (35)	−4318 (75)	4418 (22)	48
H(5)	1044 (39)	220 (77)	2384 (23)	48
H(6)	2932 (31)	2601 (72)	2165 (20)	48

* Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and hydrogen-bond parameters* (Å,°)

N(1)–C(2)	1·350 (4)	N(1)–C(6)	1·441 (3)
C(2)–N(3)	1·367 (4)	C(2)–O(2)	1·226 (3)
N(3)–C(4)	1·374 (3)	C(4)–N(5)	1·349 (4)
C(4)–O(4)	1·226 (4)	N(5)–C(6)	1·424 (5)
C(6)–O(6)	1·428 (4)	O(6)–C(7)	1·446 (3)
C(2)–N(1)–C(6)	121·9 (3)	N(1)–C(2)–N(3)	115·1 (2)
N(1)–C(2)–O(2)	122·3 (3)	N(3)–C(2)–O(2)	122·6 (3)
C(2)–N(3)–C(4)	125·1 (3)	N(3)–C(4)–N(5)	114·3 (3)
N(3)–C(4)–O(4)	121·4 (3)	N(5)–C(4)–O(4)	124·2 (2)
C(4)–N(5)–C(6)	122·9 (2)	N(1)–C(6)–N(5)	108·7 (3)
N(1)–C(6)–O(6)	107·1 (2)	N(5)–C(6)–O(6)	122·6 (2)
C(6)–O(6)–C(7)	133·5 (2)		
H(1)···O(6)′	2·09 (4)	N(1)···O(6)′	2·905 (4)
H(3)···O(2)′	2·03 (4)	N(3)···O(2)′	2·853 (4)
H(5)···O(4)′	2·04 (4)	N(5)···O(4)′	2·817 (3)
N(1)–H(1)···O(6)′	168·1 (2·3)	N(3)–H(3)···O(2)′	173·2 (2·4)
N(5)–H(5)···O(4)′	174·4 (2·4)		

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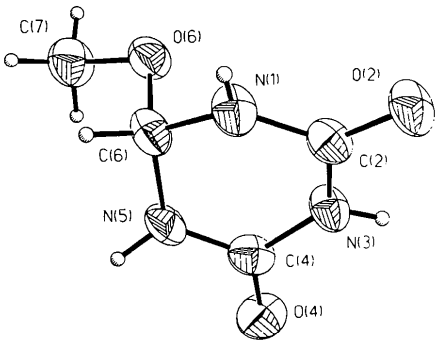

Fig. 1. Thermal-ellipsoid plot of 5-aza-6-methoxyuracil with ellipsoids drawn at 50% probability level.

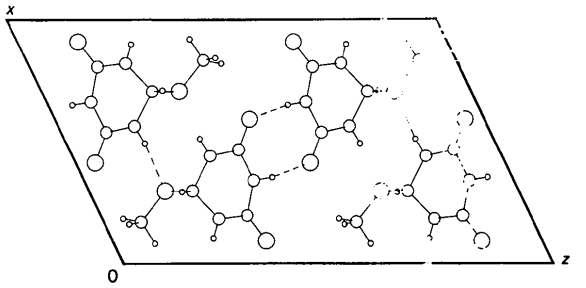

Fig. 2. Unit-cell contents viewed down **b**.

used *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0{\cdot}00030$. 106 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for H atoms, methyl-H atoms included using riding model, C—H = 0·96 Å, H—C—H = 109·5°, $U(\mathrm{H}) = 1{\cdot}2U_{eq}(\mathrm{C})$. $(\Delta/\sigma)_{max} = 0{\cdot}009$, $R = 0{\cdot}050$, $wR = 0{\cdot}058$, $S = 1{\cdot}884$. Final difference Fourier excursions 0·32 and −0·22 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).* Atom numbering for Tables 1 and 2, which give atom coordinates, bond distances and bond angles, follows that shown in Fig. 1; Fig. 2 shows the packing and hydrogen bonding.

Related literature. Structures with related ring connectivity include cyanuric acid (Verschoor & Keulen, 1971) and 2,4,6-triamino-*s*-triazine (Cromer, Larson & Stewart, 1976).

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43399 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(*m*-nitrobenzyl) Ether

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Abstract. $C_{14}H_{12}N_2O_5$, $M_r = 288{\cdot}26$, triclinic, $P\bar{1}$, $a = 7{\cdot}644$ (2), $b = 8{\cdot}378$ (2), $c = 12{\cdot}331$ (3) Å, $\alpha = 71{\cdot}96$ (2), $\beta = 74{\cdot}66$ (2), $\gamma = 63{\cdot}83$ (2)°, $U = 666{\cdot}3$ Å^3, $Z = 2$, $D_x = 1{\cdot}44$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}0$ cm^{-1}, $F(000) = 300$, $T = 293$ K, $R = 0{\cdot}049$ for 1774 reflections. The two halves of the molecule differ in the torsion angles about the O—CH_2 bonds [163·3 (3), 174·9 (3)°] and about the C—Ar bonds [−35·4 (4), 146·9 (3)° for one ring, −7·6 (4), 172·2 (3)° for the other].

Experimental. Yellow prism, 0·6 × 0·4 × 0·15 mm, by diffusion of petrol into CH_2Cl_2 solution. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode (Clegg, 1981). 2326 reflections, $2\theta_{max}$ 50°, $-h \pm k \pm l$, 2321 unique, 1774 with

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