

tionalization of the parent hydrocarbon, avoiding rearrangement between the two systems, leads to analogs of tetracyclic amine antidepressants which may be of interest (El-Zohry, Li, Tyllick & Roberts, 1988).

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Structure of a Diamino-s-tetrazine

BY RICHARD GILARDI, CLIFFORD GEORGE AND JUDITH L. FLIPPEN-ANDERSON

Laboratory for the Structure of Matter, Naval Research Laboratory, Washington, DC 20375, USA

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Abstract. 3,6-Bis(β -fluoro- β , β -dinitroethylamino)-1,2,4,5-tetrazine, $C_6H_6F_2N_{10}O_8$, $M_r = 384.17$, monoclinic, $P2_1/n$, $a = 6.569$ (2), $b = 5.821$ (2), $c = 18.153$ (6) Å, $\beta = 91.75$ (3)°, $V = 694.0$ (4) Å³, $Z = 2$, $D_x = 1.839$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71079$ Å, $\mu = 0.17$ mm⁻¹, $F(000) = 388$, $T = 225$ K, final $R = 0.051$, 1181 unique reflections. This is the first reported structural study of a diaminotetrazine. The center of the heterocyclic ring lies on a center of symmetry. Crowding in the vicinity of the nitro groups is evidenced by the eclipsed geometry [$F-C(6)-N(8)-O(8b) = 3.4$ (4), $F-C(6)-N(7)-O(7b) = -30.4$ (4)°] and close intramolecular approaches of the F atom [$F \cdots O(7b)$ at 2.537 (5) and $F \cdots O(8b)$ at 2.494 (5) Å]. Packing is influenced by the presence of an $N(4) \cdots N(2)$ intermolecular hydrogen bond [$N-H = 0.88$ (4), $H \cdots N = 2.07$ (4), $N \cdots N = 2.94$ (2) Å, and $N-H \cdots N = 169.6$ (9)°].

Experimental. Translucent orange data crystal, $0.34 \times 0.23 \times 0.44$ mm, synthesized by Robert Schmitt of SRI International, Palo Alto, California. Automated Nicolet R3m diffractometer with incident-beam graphite monochromator; 25 centered reflections within $28 \leq 2\theta \leq 35^\circ$ for determining cell parameters. Data corrected for Lorentz and polarization effects, but not for absorption. $2\theta_{\text{max}} = 50^\circ$; range of hkl : $-7 \leq h \leq 7$,

$0 \leq k \leq 6$, $0 \leq l \leq 14$; standards, $\bar{4}00$, 040, 006, monitored every 100 reflections with random variation of 3.0% over data collection, $\theta/2\theta$ mode, scan width [$2\theta(K\alpha_1) - 1.0$] to [$2\theta(K\alpha_2) + 1.0$]°, scan rate a function of count rate (6° min⁻¹ minimum, 30° min⁻¹ maximum); 1515 reflections measured, 1181 unique ($R_{\text{int}} = 0.015$), 1080 observed [$F_o > 3\sigma(F_o)$]. Structure solved by direct methods. Full-matrix least-squares refinement on F with MicroVax version of the *SHELXTL* system (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.00025$. 130 parameters refined: atom coordinates for all atoms, anisotropic temperature factors for non-H atoms and isotropic temperature factors for H atoms; $(A/\sigma)_{\text{max}} = 0.01$, $R = 0.051$, $wR = 0.089$, $S = 3.95$. Final difference Fourier excursions 0.26 and -0.29 e Å⁻³. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).* Atom numbering for Tables 1 and 2, atom coordinates, bond distances and bond angles, follows that in Fig. 1.

* 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 44960 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
F	4521 (3)	2126 (4)	809 (1)	45 (1)
N(1)	11117 (4)	567 (4)	-595 (1)	28 (1)
N(2)	10399 (4)	2215 (4)	-174 (1)	28 (1)
C(3)	9297 (5)	1603 (5)	409 (2)	24 (1)
N(4)	8614 (4)	3329 (5)	838 (1)	30 (1)
C(5)	7551 (5)	2924 (6)	1505 (2)	27 (1)
C(6)	5281 (5)	3252 (5)	1384 (2)	28 (1)
N(7)	4050 (4)	2507 (5)	2052 (2)	36 (1)
O(7a)	4971 (4)	2489 (5)	2642 (1)	52 (1)
O(7b)	2320 (5)	1988 (8)	1937 (2)	80 (1)
N(8)	4744 (4)	5802 (5)	1280 (2)	36 (1)
O(8a)	5444 (4)	7108 (4)	1744 (1)	48 (1)
O(8b)	3707 (6)	6309 (6)	750 (2)	83 (1)

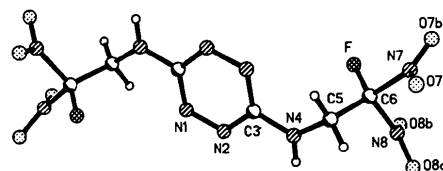


Fig. 1. Perspective drawing of the results of the X-ray study on the diaminotetrazine.

Related literature. The structures of other compounds containing nitro groups and F atoms have been reported (Koppes, Chaykovsky, Adolph, Gilardi & George, 1987). In 1,4-difluoro-1,1,4,4-tetranitro-2,3-butanediyl diacetate (Flippen-Anderson, George & Gilardi, 1988) the fluoro nitro moiety displayed a geometry similar to that found for this molecule.

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Table 2. Bond lengths (\AA) and bond angles ($^\circ$)

F—C(6)	1.321 (4)	N(1)—N(2)	1.320 (3)
N(2)—C(3)	1.346 (4)	C(3)—N(4)	1.354 (4)
C(3)—N(1')	1.337 (4)	N(4)—C(5)	1.432 (4)
C(5)—C(6)	1.514 (5)	C(6)—N(7)	1.536 (4)
C(6)—N(8)	1.537 (4)	N(7)—O(7a)	1.217 (4)
N(7)—O(7b)	1.190 (4)	N(8)—O(8a)	1.217 (4)
N(8)—O(8b)	1.203 (4)		
C(3)—N(2)—N(1)	118.0 (2)	N(4)—C(3)—N(2)	116.6 (3)
N(4)—C(3)—N(1')	119.0 (3)	N(2)—C(3)—N(1')	124.4 (3)
C(5)—N(4)—C(3)	122.6 (3)	C(6)—C(5)—N(4)	110.8 (2)
C(5)—C(6)—F	113.9 (3)	N(7)—C(6)—F	106.7 (2)
N(7)—C(6)—C(5)	112.7 (2)	N(8)—C(6)—F	107.5 (2)
N(8)—C(6)—C(5)	111.2 (3)	N(8)—C(6)—N(7)	104.2 (2)
O(7a)—N(7)—C(6)	115.9 (3)	O(7b)—N(7)—C(6)	116.9 (3)
O(7b)—N(7)—O(7a)	127.1 (3)	O(8a)—N(8)—C(6)	115.9 (3)
O(8b)—N(8)—C(6)	117.3 (3)	O(8b)—N(8)—O(8a)	126.7 (3)

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Structure of 1,2-*O*-Isopropylidene- α -D-allofuranose

BY B. SHELDRICK, W. MACKIE AND D. AKRIGG

Astbury Department of Biophysics, University of Leeds, Leeds LS2 9JT, England

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Abstract. $C_9H_{16}O_6$, $M_r = 220.2$, orthorhombic, $P2_12_12_1$, $a = 6.075$ (2), $b = 6.776$ (2), $c = 25.975$ (7) \AA , $V = 1069.2$ \AA^3 , $Z = 4$, $D_x = 1.368$ Mg m^{-3} , $\lambda(\text{Cu K}\alpha) = 1.5418$ \AA , $\mu = 0.89$ mm^{-1} , $F(000) = 472$, $T = 293$ K, $R = 0.028$ for 932 unique observed reflections [$F > 3\sigma(F)$]. The fused five-membered rings both have envelope conformations with maximum distances from the best least-squares plane for each ring of 0.159 (3) \AA for C2 and 0.209 (2) \AA for C3 and an angle between the planes of 109.8 (6) $^\circ$. The three hydroxyl groups form hydrogen bonds which make a spiral arrangement parallel to the a axis:

O(3)—HO3...O5, O5—HO5...O6 and O6—HO6...O3 with O—O distances of 3.045, 2.699 and 2.827 \AA respectively.

Experimental. The commercial sample was recrystallized from a mixture of acetone and petroleum ether. Crystal tabular, 0.50 \times 0.17 \times 0.09 mm, Enraf-Nonius CAD-4F diffractometer, Ni-filtered Cu $K\alpha$; cell parameters from 48 θ measurements in the range 19–37 $^\circ$; reflections measured for four octants to $2\theta = 140^\circ$ for ranges of h, k and l of 0 to 6, -7 to 7 and -29 to 29, respectively; 3076 reflections mea-