and size of the crystal. Final difference Fourier excursions 0·24 and −0·22 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).* Atom numbering for Tables 1 and 2 follows that shown in Fig. 1; Fig. 2 shows the packing and hydrogen bonding. Hydrogen-bond parameters are H(10)···O(18)′ = 2·17 (7), N(10)···O(18)′ = 2·88 (1) Å, and ∠N–H···O′ = 145 (5)°.

Related literature. The title compound and a closely related pentaazabicyclo[4.2.1]nonane are products of a benzylhydrazine/formaldehyde/orthoformate condensation reaction which proceeds through the intermediate compound 1,3,5-tribenzamido-1,3,5-hexahydrotriazine. For the structure of the pentaazabicyclo-[4.2.1]nonane, see Gilardi (1987). For the structure of the intermediate, see George & Gilardi (1987).

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* Lists of structure factors, anisotropic thermal parameters and hydrogen coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51744 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

GEORGE, C. & GILARDI, R. (1987). *Acta Cryst.* C**43**, 1003–1005.
GILARDI, R. (1987). *Acta Cryst.* C**43**, 1002–1003.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
SHELDRICK, G. M. (1980). *SHELXTL*80. *An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Federal Republic of Germany.

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Structure of 3-Methoxy-2,4-dinitro-2,4-diazapentane

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Abstract. $C_4H_{10}N_4O_5$, $M_r = 194{\cdot}15$, monoclinic, $P2_1/c$, $a = 7{\cdot}964$ (1), $b = 8{\cdot}320$ (1), $c = 14{\cdot}767$ (2) Å, $\beta = 119{\cdot}73$ (1)°, $V = 849{\cdot}7$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}518$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}29$ cm^{-1}, $F(000) = 408$, $T = 295$ K, final $R = 0{\cdot}044$, $wR = 0{\cdot}059$ for 1606 independent reflections. In this compound the intramolecular contacts are primarily between first-row atoms of different electronegativity. The geometry about the amino nitrogens is very nearly planar. The angle the N—N vector makes with the three-atom plane through the amino nitrogen and the adjacent carbons in the pentane chain is 1·0° at the amino nitrogen *cis* to the methoxy carbon, and 8·5° at the amino nitrogen *trans* to the methoxy carbon.

Experimental. A clear colorless 0·35 × 0·41 × 0·65 mm data crystal was provided by H. Adolph of the Naval Surface Weapons Center. Automated Nicolet *R*3*m* diffractometer with incident-beam monochromator, 25 centered reflections within $25 \le 2\theta \le 35°$ used for determining lattice parameters. $[(\sin\theta)/\lambda]_{max} = 0{\cdot}65$ Å^{-1}, range of *hkl*: $0 \le h \le 10$, $0 \le k \le 10$, $-17 \le l \le 16$. Standards (008, 040, 700) monitored every 60 reflections with random variation of 5·1% over data collection, $\theta/2\theta$ mode, scan width $[2\theta(K_{\alpha 1}) - 1{\cdot}0]$ to $[2\theta(K_{\alpha 2}) + 1{\cdot}0]°$, scan rate a function of count rate (4·0° min^{-1} minimum, 30·0° min^{-1} maximum), 2347 reflections measured, 1955 unique, $R_{int} = 0{\cdot}017$, 1606 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz and polarization but not for absorption effects. Structure solved by direct methods. The least-squares refinement used program *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/$

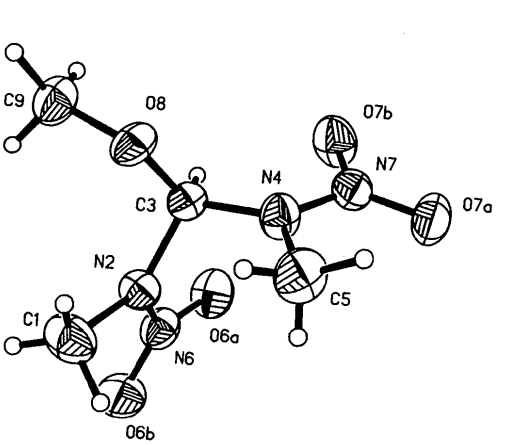

Fig. 1. Thermal ellipsoid plot of 3-methoxy-2,4-dinitro-2,4-diazapentane with ellipsoids drawn at the 20% probability level.

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$)

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

	x	y	z	U_{eq}
C(1)	1842 (6)	189 (4)	9360 (3)	66 (2)
N(2)	1695 (3)	1705 (2)	8841 (1)	43 (1)
C(3)	670 (3)	3134 (3)	8896 (2)	40 (1)
N(4)	1977 (3)	4195 (2)	9718 (1)	48 (1)
C(5)	3186 (6)	3661 (4)	10778 (2)	64 (1)
N(6)	2588 (3)	1829 (3)	8271 (2)	50 (1)
O(6*a*)	2536 (3)	3123 (2)	7865 (1)	64 (1)
O(6*b*)	3398 (3)	620 (2)	8195 (1)	71 (1)
N(7)	2265 (3)	5689 (2)	9452 (1)	49 (1)
O(7*b*)	1143 (3)	6193 (2)	8580 (1)	69 (1)
O(7*a*)	3646 (3)	6443 (2)	10122 (1)	65 (1)
O(8)	−743 (2)	2668 (2)	9125 (1)	49 (1)
C(9)	−2449 (4)	2041 (4)	8231 (2)	59 (1)

$[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.00030$. Secondary-extinction parameter $p = 0.004$ (1) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. There were 159 parameters refined: atom coordinates, anisotropic thermal parameters for all non-H atoms, isotropic thermal parameters for H atoms. $(\Delta/\sigma)_{max} = 0.004$, $R = 0.044$, $wR = 0.059$, $S = 2.164$. Final difference Fourier excursions 0·19 and −0·17 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, and bond distances and angles, respectively, follows that shown in Fig. 1.

† Lists of structure factors, anisotropic thermal parameters and hydrogen coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51749 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and bond angles* (°)

C(1)–N(2)	1·450 (4)	N(2)–C(3)	1·467 (3)
N(2)–N(6)	1·349 (4)	C(3)–N(4)	1·444 (2)
C(3)–O(8)	1·382 (4)	N(4)–C(5)	1·441 (3)
N(4)–N(7)	1·357 (3)	N(6)–O(6*a*)	1·223 (3)
N(6)–O(6*b*)	1·230 (3)	N(7)–O(7*b*)	1·221 (2)
N(7)–O(7*a*)	1·225 (2)	O(8)–C(9)	1·443 (3)
Average C–H	0·95 (6)		

C(1)–N(2)–C(3)	125·3 (3)	C(1)–N(2)–N(6)	117·9 (3)
C(3)–N(2)–N(6)	116·9 (2)	N(2)–C(3)–N(4)	110·7 (2)
N(2)–C(3)–O(8)	109·2 (2)	N(4)–C(3)–O(8)	107·5 (2)
C(3)–N(4)–C(5)	122·6 (2)	C(3)–N(4)–N(7)	118·1 (2)
C(5)–N(4)–N(7)	118·6 (2)	N(2)–N(6)–O(6*a*)	118·3 (2)
N(2)–N(6)–O(6*b*)	117·2 (2)	O(6*a*)–N(6)–O(6*b*)	124·5 (3)
N(4)–N(7)–O(7*b*)	118·7 (2)	N(4)–N(7)–O(7*a*)	116·6 (2)
O(7*b*)–N(7)–O(7*a*)	124·7 (2)	C(3)–O(8)–C(9)	112·9 (2)
Average H–C–H	108 (4)	Average *A*–*B*–H	110 (3)

Related literature. A compound with a fragment of similar connectivity with respect to the tertiary carbon is 2-chloro-5,5-dinitro-3-aza-4-oxahexene (Grigoreva, Margolis, Makarenko, Strochkina, Shchedrova, Selivanov, Melnikov & Gidaspov, 1975).

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References

GRIGOREVA, N. V., MARGOLIS, N. V., MAKARENKO, G. V., STROCHKINA, T. K., SHCHEDROVA, V. K., SELIVANOV, V. F., MELNIKOV, V. V. & GIDASPOV, B. V. (1975). *Zh. Strukt. Chem.* **16**, 157–159.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

SHELDRICK, G. M. (1980). *SHELXTL80. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Federal Republic of Germany.

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Structure of 1,9-Diacetyl-3,5,7-trinitro-1,3,5,7,9-pentaazanonane*

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(*Received* 2 *December* 1988; *accepted* 18 *January* 1989)

Abstract. $C_8H_{16}N_8O_8$, $M_r = 352.17$, monoclinic, $P2_1$, $a = 7.243$ (2), $b = 19.986$ (4), $c = 10.692$ (2) Å, $\beta = 96.18$ (3)°, $V = 1538.7$ (6) $Å^3$, $Z = 4$, $D_x = 1.520$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 11.3$ cm^{-1}, $F(000) = 736$, $T = 295$ K, final $R = 0.038$, $wR = 0.045$ for 2900 independent reflections. The two molecules in the asymmetric unit each have the same chirality and an approximate twofold axis along the N–N bond of the central nitramine in the azanonane chain. The nitrogens in the chain are roughly trigonal planar in bonding geometry with the chain torsions in the two molecules all of the same sign and ranging in magnitude from −90 to −116°; this imparts a semi-regular helicity to the chains. Intermolecular

* 5,7,9-Trinitro-3,5,7,9,11-pentaazatridecane-2,12-dione.

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