This research was supported by the National Science Foundation grants RII-8504810 for purchase of the diffractometer and RII-8610677 (EPSCoR). We thank Dr Charles L. Barnes for assistance with the measurements and helpful discussions.

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Acta Cryst. (1992). C**48**, 1679–1680

Structure of 3-Nitrato-1-nitroazetidine

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(*Received* 23 *September* 1991; *accepted* 19 *January* 1992)

Abstract. $C_3H_5N_3O_5$, $M_r = 163.1$, tetragonal, $P4_1$, $a = 10.646$ (1), $c = 5.752$ (2) Å, $V = 651.9$ (2) Å^3, $Z = 4$, $D_x = 1.662$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.15$ mm^{-1}, $F(000) = 336$, $T = 200$ K, final $R = 0.031$, $wR = 0.030$ for 415 independent observed reflections. The four-membered ring is significantly puckered, with an angle between the C—C—C and C—N—C planes of 12.7°. The amino atom N1 is pyramidal as seen by the 'out-of-plane-bend' angle between the N—N bond and the C—N—C plane, which is 39.5°. The nitro group itself shows no twist relative to the azetidine ring (average value of the four torsion angles about the N1—N1*a* bond is 0.5°).

Experimental. A clear colorless prism, 0.15 × 0.15 × 0.35 mm, data crystal was provided by Dr Kurt Baum of Fluorochem, Inc., Azusa, CA. An automated Siemens *R3m/V* diffractometer with incident-beam monochromator was used for data collection. 24 centered reflections within $13 \le 2\theta \le 35°$ were used for determining lattice parameters. $(\sin\theta/\lambda)_{max} = 0.54$ Å^{-1}, range of *hkl*: $0 \le h \le 11$, $0 \le k \le 11$, $-6 \le l \le 4$. Standards 500, 050, 004, monitored every 97 reflections, showed random variation of 3.0% over data collection: $\theta/2\theta$ scan mode, scan width $[2\theta(K\alpha_1) - 1.1]$ to $[2\theta(K\alpha_2) + 1.1]°$, ω-scan rate a function of count rate (minimum 8, maximum 15.0° min^{-1}). 544 reflections were measured, of which 482 were unique

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement coefficients* (Å^2 × 10^3)

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

	x	*y*	*z*	U_{eq}
N1	5029 (3)	1861 (3)	4851	24 (1)
N1*a*	4262 (3)	2477 (4)	6353 (10)	28 (1)
O1*a*	4073 (3)	1982 (3)	8262 (9)	30 (1)
O1*b*	3785 (3)	3467 (3)	5657 (9)	38 (1)
C2	5802 (4)	2568 (5)	3140 (11)	26 (2)
C3	6853 (4)	1611 (4)	3617 (11)	23 (2)
O3*a*	6922 (3)	813 (3)	1585 (9)	27 (1)
N3*a*	7812 (4)	−169 (4)	1873 (12)	30 (2)
O3*b*	7925 (3)	−787 (3)	131 (9)	44 (1)
O3*c*	8305 (3)	−275 (3)	3741 (9)	36 (1)
C4	6093 (5)	1055 (5)	5641 (11)	26 (2)

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

N1—N1*a*	1.358 (5)	N1—C2	1.488 (6)
N1—C4	1.491 (6)	N1*a*—O1*a*	1.234 (7)
N1*a*—O1*b*	1.237 (5)	C2—C3	1.538 (7)
C3—O3*a*	1.447 (7)	C3—C4	1.537 (8)
O3*a*—N3*a*	1.420 (5)	N3*a*—O3*b*	1.205 (8)
N3*a*—O3*c*	1.202 (8)		

N1*a*—N1—C2	120.6 (4)	N1*a*—N1—C4	122.7 (3)
C2—N1—C4	94.2 (3)	N1—N1*a*—O1*a*	117.2 (4)
N1—N1*a*—O1*b*	116.9 (5)	O1*a*—N1*a*—O1*b*	125.8 (5)
N1—C2—C3	87.1 (4)	C2—C3—O3*a*	106.4 (4)
C2—C3—C4	90.4 (4)	O3*a*—C3—C4	114.3 (4)
C3—O3*a*—N3*a*	111.8 (4)	O3*a*—N3*a*—O3*b*	111.9 (5)
O3*a*—N3*a*—O3*c*	117.7 (5)	O3*b*—N3*a*—O3*c*	130.4 (4)
N1—C4—C3	87.0 (4)		

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(R_{int} = 1.7%) and 415 were observed with $F_o > 3\sigma(F_o)$. Data were corrected for Lorentz and polarization effects, but not for absorption. The structure solution, by direct methods, and the full-matrix least-squares refinement used programs in *SHELXTL*80 (Sheldrick, 1980). The function $\sum w(|F_o| - |F_c|)^2$ was minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, g = 0.000225. H atoms were located in a difference map. 114 parameters were refined: atomic coordinates for all atoms, anisotropic thermal parameters for all non-H atoms, isotropic thermal parameters for H atoms. $(\Delta/\sigma)_{max} = 0.032$, ratio of observations to parameters = 3.6:1, $R = 0.031$ ($R = 0.041$ for all data), $wR = 0.030$, $S = 1.15$. The final difference map excursions were 0.18 and -0.15 e Å^{-3}. Atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV).* Atom numbering for Tables 1, atom coordinates, and 2, bond distances and angles, follows that shown in Fig. 1.

Related literature. This is the second example of a structural study on an N-NO_2 azetidine. The first was reported by Archibald, Gilardi, Baum & George (1990). Two structures of nitro-substituted cyclobutanes have been reported (Kai, Knochel, Kwiatkowski, Dunitz, Oth, Seebach & Kalinowski, 1982; Gilardi, George & Flippen-Anderson, 1992).

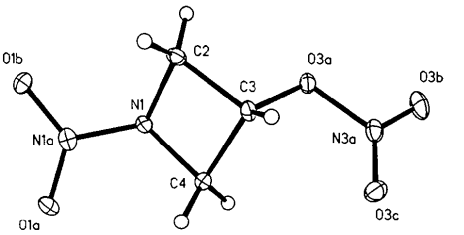

Fig. 1. A thermal ellipsoid plot of 3-nitrato-1-nitroazetidine with ellipsoids drawn at the 20% probability level.

This work was supported in part by the Office of Naval Research, Mechanics Division.

* 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 55044 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0586]

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Acta Cryst. (1992). C**48**, 1680–1681

Structure of 1,1,3,3-Tetranitrocyclobutane

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Abstract. $C_4H_4N_4O_8$, $M_r = 236.1$, triclinic, $P\bar{1}$, a = 6.301 (1), b = 7.858 (1), c = 8.736 (1) Å, α = 85.88 (1), β = 84.62 (1), γ = 85.13 (1)°, V = 428.2 (1) Å^3, $Z = 2$ (two half molecules per asymmetric unit), $D_x = 1.831$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 1.56$ mm^{-1}, $F(000) = 240$, T = 295 K, final $R = 0.031$, $wR = 0.039$ for 575 independent observed reflections. The cyclobutane rings are exactly planar (each one sits on a separate crystallographic inversion center) with the *gem*-dinitro substituents disposed symmetrically above and below the ring plane. The planes of the two nitro groups bonded to the same C atom are perpendicular to one another (dihedral angles of 89.3 and 88.4° for the two independent molecules).

Experimental. A clear colorless plate, 0.02 × 0.17 × 0.80 mm, data crystal recrystallized from methylene chloride/chloroform was provided by Dr T. Archibald of Fluorochem, Inc., Azusa, CA. An automated Siemens *R3m/V* diffractometer with incident-beam monochromator was used for data collection. 25 centered reflections within $61 \le 2\theta \le 88°$ were used for determining lattice parameters. $(\sin\theta/\lambda)_{max}$ =

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