(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).

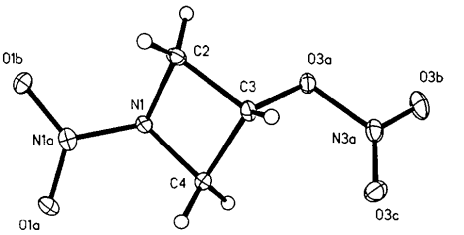

Fig. 1. A thermal ellipsoid plot of 3-nitrato-1-nitroazetidine with ellipsoids drawn at the 20% probability level.

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]

References

ARCHIBALD, T. G., GILARDI, R., BAUM, K. & GEORGE, C. (1990). *J. Org. Chem.* **55**, 2920–2924.

GILARDI, R., GEORGE, C. & FLIPPEN-ANDERSON, J. L. (1992). *Acta Cryst.* C**48**, 1680–1681.

KAI, Y., KNOCHEL, P., KWIATKOWSKI, S., DUNITZ, J. D., OTH, J. F. M., SEEBACH, D. & KALINOWSKI, H.-O. (1982). *Helv. Chim. Acta*, **65**, 137–161.

SHELDRICK, G. M. (1980). *SHELXTL*80. *An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Germany.

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. $(\sin\theta/\lambda)_{max}$ =

0108-2701/92/091680-02$06.00

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

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

	x	y	z	U_{eq}
Cl	−511 (8)	−170 (8)	1225 (8)	27 (1)
N1	672 (4)	−992 (3)	2488 (3)	40 (1)
O1	1752 (6)	−129 (5)	3126 (4)	68 (1)
O2	490 (5)	−2511 (4)	2782 (3)	60 (1)
N2	−2694 (4)	476 (3)	1912 (4)	41 (1)
O3	−3919 (4)	1123 (3)	996 (4)	68 (1)
O4	−3080 (16)	335 (18)	3316 (12)	64 (2)
C2	615 (7)	1225 (4)	187 (3)	34 (1)
C1′	−5234 (8)	−5095 (9)	3838 (8)	29 (2)
N1′	−3853 (4)	−4435 (3)	2418 (3)	36 (1)
O1′	−2455 (16)	−5421 (17)	1925 (12)	55 (2)
O2′	−4260 (5)	−2988 (4)	1923 (3)	55 (1)
N2′	−7089 (4)	−5808 (3)	3179 (3)	39 (1)
O3′	−8378 (5)	−6373 (3)	4167 (4)	68 (1)
O4′	−7164 (5)	−5803 (5)	1823 (4)	60 (1)
C2′	−4082 (8)	−6258 (4)	4988 (3)	36 (1)

0.56 $Å^{-1}$, range of *hkl*: $-7 \le h \le 0$, $-8 \le k \le 8$, $-9 \le l \le 9$. Standards $\bar{2}1\bar{2}$, $\bar{1}13$, $0\bar{4}2$, monitored every 97 reflections, showed random variation of 2.0% over data collection; $\theta/2\theta$ scan mode, scan width $[2\theta(K\alpha_1) - 1.0]$ to $[2\theta(K\alpha_2) + 1.0]°$, ω-scan rate a function of count rate (minimum 2.0, maximum 15.0° min^{-1}). 739 reflections were measured, of which 639 were unique ($R_{int} = 0.60\%$) and 575 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. 162 parameters were refined: atomic coordinates for all atoms, anisotropic thermal parameters for all non-H atoms, isotropic thermal parameters for H atoms. $(\Delta/\sigma)_{max} = 0.01$ (for non-H atoms, 0.80 for H atoms), ratio of observations to parameters = 3.5:1, $R = 0.031$ ($R = 0.035$ for all data), $wR = 0.039$, $S = 1.98$. The final difference map excursions were 0.13 and -0.12 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. The first syntheses of *gem*-dinitro cyclobutane compounds, including the title compound, were reported by Archibald, Garver, Baum & Cohen (1989). Only one other nitro-substituted cyclobutane crystal structure has been reported: 1,1′-dinitrobicyclobutyl (Kai, Knochel, Kwiatkowski, Dunitz, Oth, Seebach & Kalinowski, 1982). The structure of a related molecule, 1,3,3-trinitroazetidine has also been reported (Archibald, Gilardi, Baum & George, 1990).

* 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 55043 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0587]

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

Atoms labelled with an asterisk are related by the inversion center at the center of the ring to a similarly labeled atom of the asymmetric unit.

Cl—N1	1.470 (7)	Cl—N2	1.510 (6)
Cl—C2	1.547 (7)	Cl—C2*	1.545 (8)
N1—O1	1.200 (5)	N1—O2	1.215 (4)
N2—O3	1.219 (4)	N2—O4	1.227 (11)
C1′—N1′	1.532 (7)	C1′—N2′	1.515 (7)
C1′—C2′	1.505 (7)	C1′—C2′*	1.538 (8)
N1′—O1′	1.193 (11)	N1′—O2′	1.203 (4)
N2′—O3′	1.215 (4)	N2′—O4′	1.190 (4)

N1—C1—N2	107.6 (4)	N1—C1—C2	116.4 (4)
N2—C1—C2	112.4 (4)	N1—C1—C2*	116.8 (4)
N2—C1—C2*	112.8 (4)	C2—C1—C2*	90.3 (4)
C1—N1—O1	117.9 (4)	C1—N1—O2	116.6 (4)
O1—N1—O2	125.4 (3)	C1—N2—O3	115.7 (4)
C1—N2—O4	118.5 (6)	O3—N2—O4	125.7 (6)
C1—C2—C1′	89.7 (4)	N1′—C1′—N2′	104.2 (5)
N1′—C1′—C2′	115.9 (4)	N2′—C1′—C2′	116.3 (5)
N1′—C1′—C2′*	114.0 (5)	N2′—C1′—C2′*	113.8 (4)
C2′—C1′—C2′*	92.9 (4)	C1′—N1′—O1′	115.9 (6)
C1′—N1′—O2′	117.6 (3)	O1′—N1′—O2′	126.6 (6)
C1′—N2′—O3′	112.9 (4)	C1′—N2′—O4′	120.6 (3)
O3′—N2′—O4′	126.5 (3)	C1′—C2′—C1*	87.1 (4)

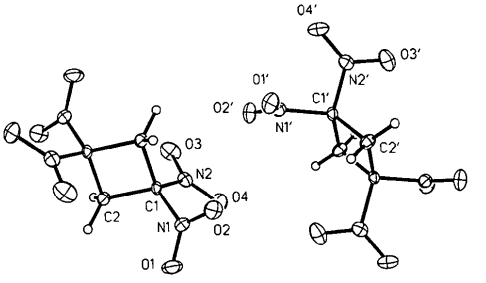

Fig. 1. A thermal ellipsoid plot of 1,1,3,3-tetranitrocyclobutane with ellipsoids drawn at the 20% probability level. The asymmetric unit consists of two half molecules.

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

References

ARCHIBALD, T. G., GARVER, L. C., BAUM, K. & COHEN, M. C. (1989). *J. Org. Chem.* **54**, 2869–2873.

ARCHIBALD, T. G., GILARDI, R., BAUM, K. & GEORGE, C. (1990). *J. Org. Chem.* **55**, 2920–2924.

KAI, Y., KNOCHEL, P., KWIATKOWSKI, S., DUNITZ, J. D., OTH, J. F. M., SEEBACH, D. & KALINOWSKI, H.-O. (1982). *Helv. Chim. Acta*, **65**, 137–161.

SHELDRICK, G. M. (1980). *SHELXLTL*80. *An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. Univ. of Göttingen, Germany.