

Fig. 2. Molecular packing viewed along *c* showing half of the unit cell. The H...Cl<sup>-</sup> distances of the two N-H...Cl hydrogen bonds are indicated by dashed lines. The short H...Cl<sup>-</sup> contact H(14)B...Cl and the short intramolecular H...H contact H(16)A...H(20)B are indicated by dotted lines. C, N and Cl atoms are represented by small filled, small open, and large open circles respectively. Distances in Å.

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### *N,N'*-Dinitrosopentamethylenetetramine, \*C<sub>5</sub>H<sub>10</sub>N<sub>6</sub>O<sub>2</sub>

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**Abstract.**  $M_r = 186.18$ , monoclinic,  $P2_1/a$ ,  $a = 12.156$  (8),  $b = 7.397$  (5),  $c = 9.923$  (7) Å,  $\beta = 113.8$  (1)°,  $V = 816.38$  Å<sup>3</sup>,  $D_x = 1.515$ ,  $D_m = 1.525$  Mg m<sup>-3</sup>,  $Z = 4$ ,  $F(000) = 392$ ,  $\lambda(\text{Cu } K\alpha) = 1.5418$ ,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu(\text{Cu } K\alpha) = 1.04$  mm<sup>-1</sup>,  $\mu(\text{Mo } K\alpha) = 0.13$  mm<sup>-1</sup>,  $R = 0.057$  for 955 observed [ $I > 3\sigma(I)$ ] reflexions. The structure comprises discrete molecules with no intermolecular interactions other than van der Waals forces.

**Introduction.** The title compound (Fig. 1) is marketed under the trade-name 'Vulcacer BN' and is used as a blowing agent in the production of microcellular materials, e.g. 'crepe' or 'sponge' rubber. The structural analysis was undertaken to ascertain whether any relationship exists between the nitroso-group dimensions and the ease of evolution of gaseous N and O during processing, and the occasionally observed instability of certain mixes involving the compound.

**Experimental.** Prismatic crystals (m.p. 480 K, decomp.) of the title compound prepared by Dr R. A. Darrall of

\* IUPAC name: 3,7-dinitroso-1,3,5,7-tetraazabicyclo[3.3.1]nonane.

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Imperial Chemical Industries Limited (Organics Division) by reacting hexamethylenetetramine with nitrous acid and recrystallizing the product from absolute ethanol; unit-cell dimensions obtained initially from rotation photographs and more precisely from a least-squares fit of  $2\theta$  angles on a Stoe STADI-2 diffractometer; intensities obtained from multiple-films on Weissenberg layers  $h0l \leftarrow h7l$  and  $hk0$  using filtered Cu radiation, supplemented by  $hk0 \rightarrow hk11$  layers on the diffractometer using graphite-monochromated Mo radiation with  $\omega$  scans; corrections applied for  $L_p$  effects but not for absorption, or extinction; index range  $h 0 \rightarrow 14$ ,  $k 0 \rightarrow 7$ ,  $l -11 \rightarrow 11$ ; structure solved using direct methods in the NRC programs (Ahmed, Hall, Pippy & Huber, 1970) modified for use on the ICL 1905E computer at the London Polytechnics Computer Unit. The  $E$  map resulting from 227 signed  $E$  values ( $E > 1.5$ ) gave all the non-H atoms; parameters refined by least-squares using initially  $B_{iso}$  and  $\sqrt{w} = 1/F_o$ , and finally  $B_{ij}$ ; calculated H positions included in structure factor calculations with  $B_{iso} = 5.0$  Å<sup>2</sup>, but not refined; indicated shifts all  $< 0.2\sigma$ ; scattering factors from *International Tables for X-ray Crystallography* (1962);  $R_w = 0.119$ .

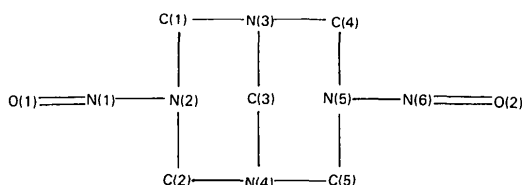


Fig. 1. Schematic chemical formula.

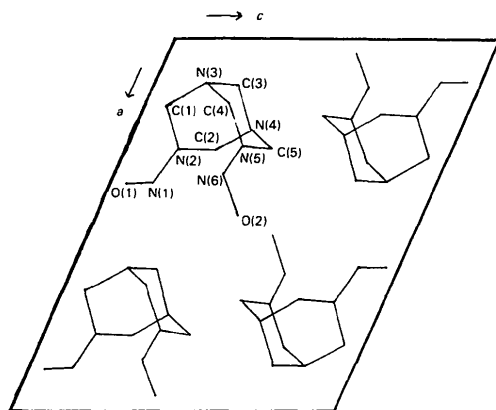


Fig. 2. Numbering of atoms and packing of molecules in (010) projection.

**Discussion.** Final atomic parameters are listed in Table 1.\* Numbering of atoms and the packing of the molecules in the unit cell, (010) projection, are shown in Fig. 2. Bond lengths and interbond angles are in Table 2. The average C—N distance is 1.452 Å compared with 1.478 Å in hexamethylenetetramine (Becka & Cruickshank, 1963), and the mean internal 'tetrahedral' angle at C is 110.6° compared with 107.4° in hexamethylenetetramine. The N=O mean length is 1.160 Å for which Pauling (1944) predicted 1.18 Å. Other comparable values are 1.199 Å in *N*-acetyl-*S*-nitroso-DL-penicillamine (Carnahan, Lenhart & Ravichandran, 1978) and 1.206 Å in *N*-nitrosodiphenylamine (Banerjee, Brown & Lewis, 1982), indicating the possibility of a bond order greater than 2 in this compound; most other nitroso compounds have lengths more than 1.20 Å.

There seems to be no explanation of the difference between the two N=O bond lengths, 1.179 and 1.141 Å; both O atoms are involved in contacts across

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38579 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final atomic positional and isotropic thermal parameters

$$B_{\text{eq}} = \frac{4}{3} \left( \frac{\beta_{11}}{a^{*2}} + \frac{\beta_{22}}{b^{*2}} + \frac{\beta_{33}}{c^{*2}} \right).$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub> (Å <sup>2</sup> )
C(1)	0.1726 (3)	-0.0005 (5)	0.0603 (4)	3.40 (6)
C(2)	0.3038 (4)	0.1953 (5)	0.2677 (4)	3.67 (6)
C(3)	0.1190 (4)	0.0795 (6)	0.2566 (5)	4.49 (7)
C(4)	0.1654 (4)	-0.2283 (5)	0.2360 (4)	3.89 (7)
C(5)	0.2977 (4)	-0.0359 (5)	0.4428 (5)	3.98 (7)
N(1)	0.3972 (3)	0.0216 (4)	0.1395 (4)	4.83 (7)
N(2)	0.2896 (2)	0.0712 (4)	0.1479 (3)	3.27 (5)
N(3)	0.1150 (2)	-0.0679 (4)	0.1555 (3)	2.90 (5)
N(4)	0.2417 (3)	0.1224 (4)	0.3536 (3)	3.44 (5)
N(5)	0.2829 (2)	-0.1964 (4)	0.3497 (3)	3.01 (5)
N(6)	0.3711 (4)	-0.3154 (8)	0.3425 (5)	6.54 (9)
O(1)	0.3885 (3)	-0.0761 (5)	0.0419 (4)	6.45 (8)
O(2)	0.4625 (4)	-0.2693 (5)	0.4272 (4)	6.47 (8)

Table 2. Bond lengths (Å) and interbond angles (°)

C(1)—N(2)	1.436 (5)	N(2)—C(1)—N(3)	110.4 (3)
C(1)—N(3)	1.471 (5)	N(2)—C(2)—N(4)	109.1 (3)
C(2)—N(2)	1.456 (5)	N(3)—C(3)—N(4)	111.0 (3)
C(2)—N(4)	1.451 (6)	N(3)—C(4)—N(5)	111.7 (3)
C(3)—N(3)	1.469 (5)	N(4)—C(5)—N(5)	111.0 (3)
C(3)—N(4)	1.447 (6)	N(2)—N(1)—O(1)	115.9 (4)
C(4)—N(3)	1.423 (5)	C(1)—N(2)—C(2)	119.3 (3)
C(4)—N(5)	1.439 (5)	C(1)—N(2)—N(1)	126.2 (3)
C(5)—N(4)	1.460 (5)	C(2)—N(2)—N(1)	114.2 (3)
C(5)—N(5)	1.470 (5)	C(1)—N(3)—C(3)	107.1 (3)
N(1)—N(2)	1.392 (6)	C(1)—N(3)—C(4)	115.7 (3)
N(5)—N(6)	1.411 (6)	C(3)—N(3)—C(4)	110.3 (3)
N(1)—O(1)	1.179 (6)	C(2)—N(4)—C(3)	109.3 (3)
N(6)—O(2)	1.141 (7)	C(2)—N(4)—C(5)	115.4 (3)
		C(3)—N(4)—C(5)	109.3 (3)
		C(4)—N(5)—C(5)	117.1 (3)
		C(4)—N(5)—N(6)	113.1 (4)
		C(5)—N(5)—N(6)	129.3 (4)
		N(5)—N(6)—O(2)	108.1 (5)

centres of symmetry, O(1)···O(1') = 3.335 (6) and O(2)···O(2') = 3.675 (5) Å. Other close intermolecular approaches are O(2)···C(2') (centre-of-symmetry related) 3.255 (6) Å, and O(1)···C(1') = 3.291 (5) and O(1)···C(4') = 3.450 (6) Å, both screw-axis related.

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