Acta Cryst. (1990). C46, 1959–1960

Structure of the Diammonium Salt of 4,4′,5,5′-Tetranitro-2,2′-biimidazole, $C_6N_8O_8.2NH_4$*

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(*Received* 3 *October* 1989; *accepted* 2 *April* 1990)

Abstract. $C_6H_8N_{10}O_8$, $M_r = 348{\cdot}19$, monoclinic, $P2_1/c$, $a = 3{\cdot}939\ (2)$, $b = 9{\cdot}153\ (2)$, $c = 17{\cdot}822\ (3)$ Å, $\beta = 94{\cdot}27\ (3)°$, $V = 640{\cdot}8$ Å^3, $Z = 2$, $D_x = 1{\cdot}805$ Mg m^{-3}, λ(Mo $K\alpha_1$) $= 0{\cdot}70926$ Å, $\mu = 0{\cdot}16$ mm^{-1}, $F(000) = 356$, room temperature, final $R = 0{\cdot}035$ for 763 observed reflections $I > 2\sigma(I)$ out of 1129 independent reflections. The molecule lies on a center of symmetry. All H atoms take part in hydrogen bonding to produce a rigid network. Bond lengths are normal.

Experimental. Title compound prepared by neutralizing a solution of tetranitrobiimidazole with concentrated aqueous ammonia. Crystals grown by evaporation of resulting solution. Selected crystal *ca* $0{\cdot}40 \times 0{\cdot}12 \times 0{\cdot}10$ mm. CAD-4 diffractometer, θ–2θ scan. Scan range $(1{\cdot}0 + 0{\cdot}34\tan\theta)°$. Scan speed 1·5 to 3·3° θ min^{-1}. Background first and last 1/6 of scan. Graphite-monochromated Mo $K\alpha$ radiation. Unit cell, 25 reflections $10 < \theta < 20°$. No absorption corrections. $[(\sin\theta)/\lambda]_{max} = 0{\cdot}596$ Å^{-1}. Index range $-4 \le h \le 4$, $0 \le k \le 10$, $-20 \le l \le 20$, 2252 reflections measured and averaged to yield 1129 unique reflections of which 763 were observed with $I > 2\sigma(I)$, $R_{int} = 0{\cdot}020$. Standard reflections 206 and 137 showed no significant variation. Least squares minimized $\sum w(\Delta F)^2$ with $w = [\sigma_c^2(F) + 0{\cdot}02F^2]^{-1}$, $\sigma_c^2(F)$ based on counting statistics. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971), H atoms by difference Fourier synthesis. 125 parameters including the scale factor, positional parameters, anisotropic thermal parameters for C, N, O, and isotropic thermal parameters for H were refined. Final $R = 0{\cdot}035$, $wR = 0{\cdot}034$, $S = 1{\cdot}7$. Max. $\Delta/\sigma = 5 \times 10^{-3}$. Final ΔF Fourier synthesis $-0{\cdot}22 < \Delta\rho < 0{\cdot}21$ e Å^{-3}. Scattering factors f (RHF for C, N, O and SDS for H), f', f'' from *International Tables for X-ray Crystallography* (1974). Calculations on CRAY-1 using the Los Alamos crystal structure system developed primarily by A. C. Larson.

Fig. 1 is an *ORTEP* (Johnson, 1965) drawing of the molecule showing the atom-numbering scheme. Final parameters are given in Table 1.‡ Bond lengths and angles are given in Table 2. Stereo drawing shown in Fig. 2. The asymmetric unit is 1/2 of the

* This work was performed under the auspices of the US Department of Energy.

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‡ Lists of structure factors, ansitropic thermal parameters, H-atom parameters and bond lengths involving hydrogen have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52914 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

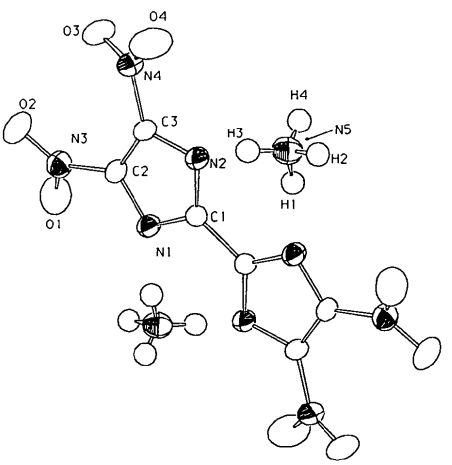

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule to show atom-numbering scheme. Thermal ellipsoids are 30% probability. H-atom size is arbitrary.

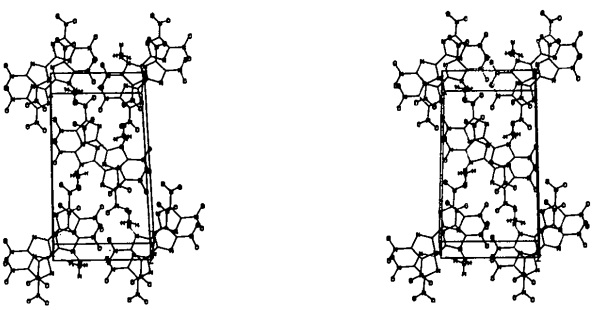

Fig. 2. Stereo drawing of the structure. The origin is at the lower left rear, with z vertical and y horizontal. Hydrogen bonds are dotted.

0108-2701/90/101959-02$03.00

Table 1. *Final least-squares parameters for* C, N *and* O *in the diammonium salt of tetranitrobiimidazole* ($\times 10^4$) *and equivalent isotropic U values* ($\times 10^2$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq} (Å^2)
C(1)	5115 (6)	5547 (2)	4709 (1)	2·4 (2)
C(2)	6067 (6)	6516 (3)	3677 (1)	2·7 (2)
C(3)	4840 (6)	7560 (3)	4145 (1)	2·8 (2)
N(1)	6248 (5)	5220 (2)	4035 (1)	2·8 (2)
N(2)	4242 (5)	6951 (2)	4811 (1)	2·8 (2)
N(3)	6996 (6)	6608 (2)	2916 (1)	3·5 (2)
N(4)	4322 (7)	9105 (2)	4043 (1)	4·1 (3)
O(1)	9050 (5)	5723 (2)	2709 (1)	5·4 (2)
O(2)	5675 (6)	7564 (2)	2507 (1)	5·3 (2)
O(3)	5912 (6)	9742 (2)	3579 (1)	6·3 (3)
O(4)	2352 (7)	9708 (2)	4440 (1)	7·2 (3)
Ammonium ion				
N(5)	10054 (8)	2558 (3)	3829 (2)	3·9 (3)

$C_6N_8O_8^{2-}$ anion and one NH_4^+ cation. The rings are planar within 0·03 Å. The nitro groups are twisted out of the ring plane by 18·9 (1) and 21·6 (1)°. The ammonium ion is hydrogen bonded to four different molecules. Two H atoms make hydrogen bonds to ring N atoms and two to O atoms. This rigid structure is compatible with the relatively high decomposition temperature (without melting) of 523–533 K. Bond distances are very similar to those found in biimidazole (Cromer, Ryan & Storm, 1987) and in tetranitrobiimidazole dihydrate (Cromer & Storm, 1990).

Related literature. References to structures of other small high-energy molecules are given by Cromer, Hall, Lee & Ryan (1988).

Table 2. *Bond lengths* (Å) *and angles* (°) *in the diammonium salt of tetranitrobiimidazole*

C(1)—C(1)	1·451 (1)	C(3)—N(2)	1·347 (3)
C(1)—N(1)	1·346 (3)	C(3)—N(4)	1·438 (3)
C(1)—N(2)	1·345 (3)	N(3)—O(1)	1·221 (3)
C(2)—N(1)	1·346 (3)	N(3)—O(2)	1·229 (3)
C(2)—N(3)	1·434 (3)	N(4)—O(3)	1·223 (3)
C(2)—C(3)	1·379 (3)	N(4)—O(4)	1·220 (3)

C(1)—C(1)—N(1)	121·9 (1)	C(1)—N(1)—C(2)	102·5 (2)
C(1)—C(1)—N(2)	122·2 (1)	C(1)—N(2)—C(3)	102·3 (2)
N(1)—C(1)—N(2)	116·0 (2)	C(3)—N(3)—O(1)	118·0 (2)
C(3)—C(2)—N(1)	109·5 (2)	C(3)—N(3)—O(2)	118·4 (2)
C(3)—C(2)—N(3)	131·1 (2)	O(1)—N(3)—O(2)	123·6 (2)
N(1)—C(2)—N(3)	119·4 (2)	C(2)—N(4)—O(3)	118·6 (3)
C(2)—C(3)—N(2)	109·7 (2)	C(2)—N(4)—O(4)	117·5 (2)
C(2)—C(3)—N(4)	131·1 (2)	O(3)—N(4)—O(4)	123·9 (2)
N(2)—C(3)—N(4)	119·1 (2)		

Hydrogen bonds

X—H···Y	Symmetry operation on Y	$d(X—Y)$	d(H···Y)	∠X—H···Y
N(5)—H(1)···N(1)	x, y, z	2·898 (3)	2·00 (4)	173 (3)
N(5)—H(3)···N(2)	$2-x, 1-y, 1-z$	3·091 (3)	2·36 (6)	160 (5)
N(5)—H(2)···O(2)	$2-x, -\frac{1}{2}+y, \frac{1}{2}-z$	3·017 (3)	2·04 (6)	177 (6)
N(5)—H(4)···O(3)	$x, -1+y, z$	3·065 (4)	2·35 (5)	168 (6)

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Acta Cryst. (1990). C**46**, 1960–1962

Structure of *tert*-Butyl 4-Aminomethyl-3-carboxymethyl-2,3,4-trideoxy-5-hydroxymethyl-α-D-lyxopyranoside-δ-lactam

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(*Received* 30 *November* 1989; *accepted* 27 *March* 1990)

Abstract. $C_{13}H_{23}NO_4.H_2O$, $M_r = 275·34$, monoclinic, $C2$, $a = 15·725$ (6), $b = 6·871$ (2), $c = 14·100$ (7) Å, $\beta = 93·70$ (3)°, $V = 1520·3$ Å^3, $Z = 4$, $D_x = 1·203$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ(Cu $K\alpha$) = 0·721 mm^{-1}, $F(000) = 600$, $T = 293$ K, $R = 0·047$, $wR = 0·046$ for 1511 observed reflections. The title compound contains a bicyclic α-D-pyranoside system that has a *cis* ring junction with the δ-lactam ring. The molecules are held in the crystal by hydrogen bonds of the O—H···O type or N—H···O type. The O···O distances range from 2·707 (3) to 2·875 (4) Å, while the N···O distance is 2·876 (4) Å.

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0108-2701/90/101960-03$03.00