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endo-3,8-Dioxatricyclo[3.2.1.$0^{2,4}$]octane-6-carbonitrile

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Abstract. $C_7H_7NO_2$, $M_r = 137{\cdot}14$, $P2_1/c$, monoclinic, $a = 8{\cdot}572\ (4)$, $b = 8{\cdot}693\ (4)$, $c = 11{\cdot}706\ (5)$ Å, $\beta = 131{\cdot}93\ (6)°$, $V = 649\ (2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}41\ (3)$, $D_x = 1{\cdot}40$ Mg m^{-3}, Cu $K\bar{\alpha}$, $\lambda = 1{\cdot}5418$ Å, $\mu = 0{\cdot}88$ mm^{-1}, $F(000) = 288$, $T = 293\ (2)$ K, final $R = 0{\cdot}082$ for 979 observed reflections. The boat conformation of the six-membered ring is not distorted by the cyano group, the dihedral angles C(3)—C(4)—C(5)—C(6) and C(3)—C(2)—C(1)—C(6) being equal to 0·0 (5)°. Short intermolecular distances are observed between the H atoms of the molecule and the nucleophilic O and N atoms of the surrounding molecules. All the H atoms except H(51) and H(52) are electrophilic. These interactions are responsible for packing, the density of this compound being significantly higher than the density ($\simeq 1{\cdot}1$ Mg m^{-3}) of other cyano compounds [Viani, Cossu & Lapasset (1981). *Acta Cryst.* B**37**, 484–486].

Experimental. The two stereoisomers (*endo* and *exo*) were prepared by epoxidation of 5-cyano-7-oxabicyclo[2.2.1]hept-2-ene isomers (Kienzle, 1975) and separated by thin-layer chromatography. Crystal size 0·21 × 0·35 × 0·36 mm. D_m measured by flotation. Enraf–Nonius CAD-3 diffractometer used in the θ–2θ scan mode. No absorption correction. $P2_1/c$ symmetry and cell parameters determined by preliminary Weissenberg pictures. Standard reflection: 132 stable throughout experiment (intensity variation < 8%), monitored after every 60 reflections. 1106 measured reflections and 979 observed with $I > 2{\cdot}5\sigma(I)$. h 0–15, k 0–11, l −15–15. $\theta_{\max} = 65°$. Structure solved by direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The E map based on phases corresponding to the best figure of merit yielded all the non-H atoms. Structure refined by full-matrix least squares with *SHELX*76 (Sheldrick, 1976), $\sum w(\Delta F)^2$ minimized. All the H atoms revealed by difference Fourier synthesis were introduced in the

Table 1. *Final fractional coordinates* (× 10^4) *and isotropic thermal parameters for the non-*H *atoms with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
C(1)	4794 (4)	3160 (3)	−2381 (3)	4·7 (2)
C(2)	4988 (4)	3026 (3)	−1066 (3)	4·4 (2)
C(3)	7148 (4)	3719 (3)	230 (3)	4·4 (2)
C(4)	8750 (4)	2494 (3)	592 (3)	4·5 (2)
C(5)	8500 (4)	2634 (3)	−845 (3)	4·9 (2)
C(6)	6830 (5)	3912 (3)	−1735 (3)	4·8 (2)
O(7)	7335 (3)	4855 (2)	−537 (2)	5·1 (2)
O(8)	3538 (3)	4111 (2)	−2249 (2)	5·6 (2)
C(9)	8376 (4)	941 (3)	848 (3)	4·6 (2)
N(10)	8064 (4)	−270 (3)	1031 (3)	5·7 (2)

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Table 2. *Interatomic distances* (Å) *and angles* (°) *involving non*-H *atoms with e.s.d.'s in parentheses*

C(1)–C(2)	1·438 (6)	C(3)–O(7)	1·414 (4)
C(1)–C(6)	1·511 (5)	C(4)–C(5)	1·552 (6)
C(1)–O(8)	1·444 (5)	C(4)–C(9)	1·464 (4)
C(2)–C(3)	1·529 (3)	C(5)–C(6)	1·541 (4)
C(2)–O(8)	1·433 (3)	C(6)–O(7)	1·419 (4)
C(3)–C(4)	1·555 (4)	C(9)–N(10)	1·141 (4)

C(2)–C(1)–C(6)	103·3 (2)	C(3)–C(4)–C(9)	114·4 (3)
C(2)–C(1)–O(8)	59·6 (2)	C(5)–C(4)–C(9)	113·7 (2)
C(6)–C(1)–O(8)	113·1 (2)	C(4)–C(5)–C(6)	100·5 (3)
C(1)–C(2)–C(3)	103·1 (3)	C(1)–C(6)–C(5)	105·5 (2)
C(1)–C(2)–O(8)	60·4 (2)	C(1)–C(6)–O(7)	103·4 (4)
C(3)–C(2)–O(8)	113·1 (2)	C(5)–C(6)–O(7)	102·4 (2)
C(2)–C(3)–C(4)	105·3 (2)	C(3)–O(7)–C(6)	97·8 (2)
C(2)–C(3)–O(7)	102·8 (2)	C(1)–O(8)–C(2)	60·0 (2)
C(4)–C(3)–O(7)	101·4 (3)	C(4)–C(9)–N(10)	179·3 (2)
C(3)–C(4)–C(5)	101·2 (2)		

refinement procedure but not refined with the B_{eq}'s of the atoms to which they were bonded. Weighting scheme $w = 1/[\sigma^2(F) + 0{\cdot}063183F^2]$, $R = 0{\cdot}082$, $wR = 0{\cdot}085$. High R value explained by the poor quality of crystalline sample. $(\Delta/\sigma)_{max} = 0{\cdot}270$, $(\Delta/\sigma)_{min} = 0{\cdot}001$. $(\Delta/\rho)_{max} = 0{\cdot}32$ e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). All numerical calculations were performed on the 370/145 IBM computer of the Office Central de Mécanographie of Abidjan. Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 shows the molecular structure and numbering scheme.

Related literature. The present research was initiated to confirm the postulated *endo* position of the cyano group in the first compound isolated by Marfisi, Cossu & Aycard (1981). The structure may be compared to those of 6-chloro-6-cyano-1,5-dimethyl-3,8-dioxatricyclo[3.2.1.0^{2,4}]octane (Cossu, Viani, Lapasset, Aycard, Marfisi & Bodot, 1982) and related cyano compounds (Viani, Cossu & Lapasset, 1981).

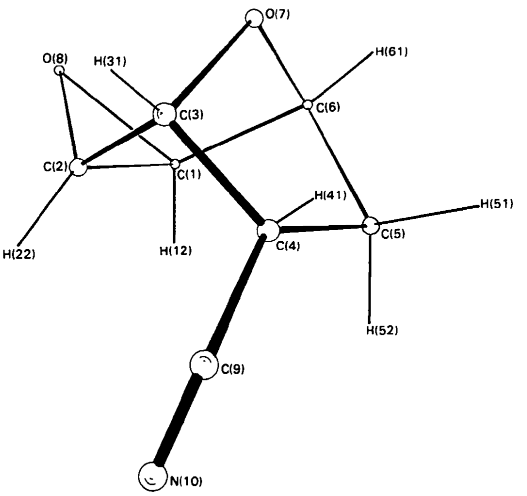

Fig. 1. The molecular structure with the numbering scheme.

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* Lists of structure factors, torsion angles and shortest intermolecular contacts have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43229 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1,2,3-Triaminoguanidine (TAG)

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Abstract. CH_8N_6, $M_r = 104{\cdot}2$, monoclinic, $P2_1/c$, $a = 7{\cdot}460$ (3), $b = 10{\cdot}274$ (2), $c = 6{\cdot}343$ (3) Å, $\beta = 110{\cdot}80$ (2)°, $V = 454{\cdot}5$ Å^3, $Z = 4$, $D_x = 1{\cdot}52$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 10{\cdot}1$ cm^{-1}, $F(000) = 224$, $T = 168$ (1) K, final $R = 0{\cdot}031$ for 771 unique observed reflections. One C—N distance [1·292 (1) Å] in the TAG molecule is significantly short; all other bond distances fall within the accepted ranges for C—N and

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